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MANUFACTURING METHODS AND TECHNOLOGY MEASURE FOR MICROWAVE FERR--ETC(U)

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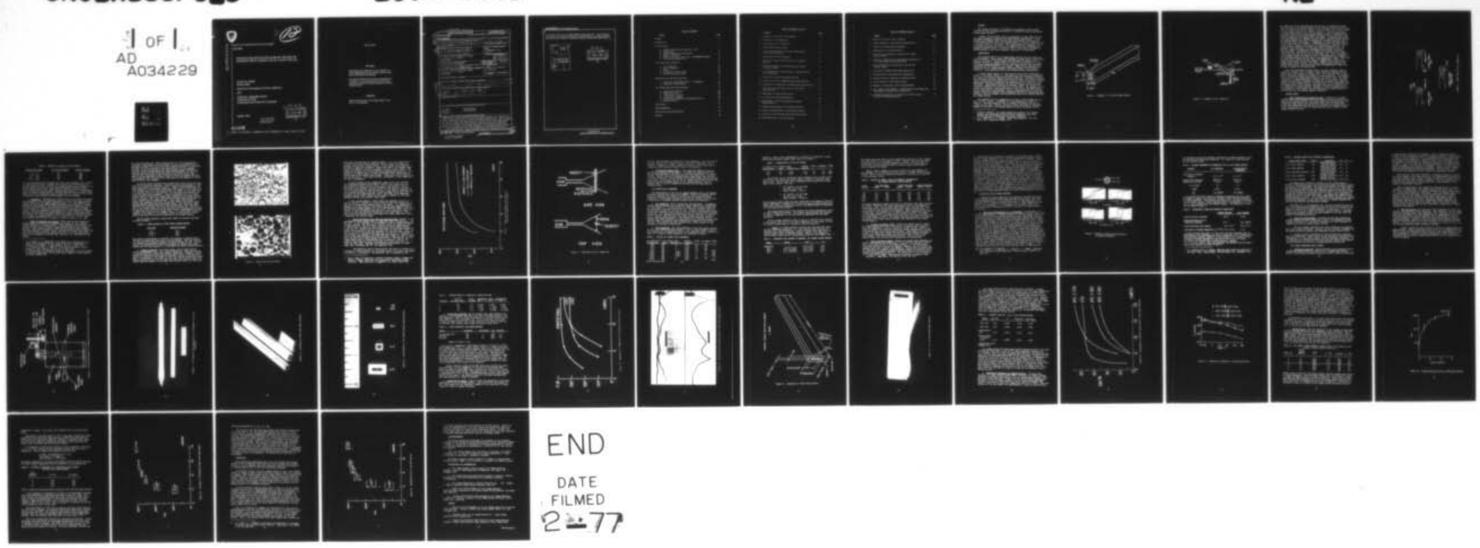

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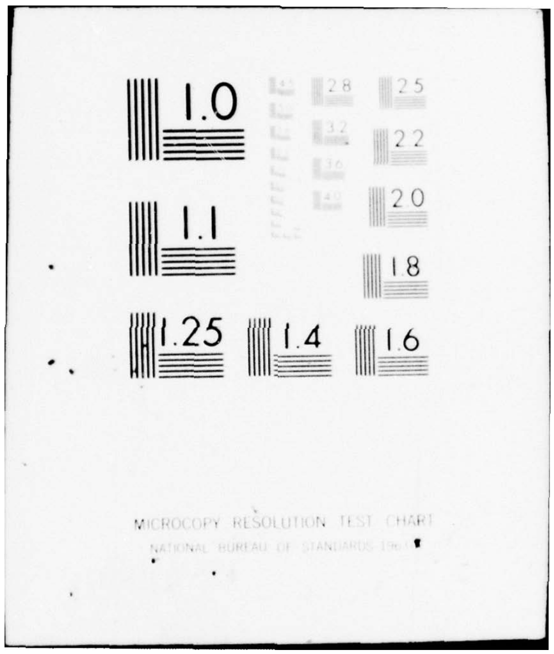
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Research and Development Technical Report

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MANUFACTURING METHODS AND TECHNOLOGY MEASURE FOR MICROWAVE FERRITE POWDERS FOR ARC PLASMA SPRAYING

Richard W. Babbitt
William Wade

Electronics Technology and Devices Laboratory

FOR

Production Integration Branch
Production Division
Procurement and Production Directorate

October 1976

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Commercially available spray-dried lithium ferrite powders were selected for the arc plasma fabrication of C-Band and S-Band phase shifters. Several spray-dried powders were evaluated and only those which flowed freely were suitable for the arc plasma spray (APS) process. Selected spray-dried powders were sprayed over a range of arc plasma conditions and the magnetic properties measured to determine the best APS parameters for fabricating phase shifters.

The necessary ovens and control equipment were designed and built for spraying phase shifters. Several different dielectric compositions which were

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to be used as inserts for phase shifters were evaluated. Non-reciprocal, waveguide, ferrite phase shifters were fabricated by arc plasma spraying, and the devices tested for insertion loss and differential phase shift.

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PURPOSE

The primary objective is to establish an economical ferrite powder suitable for the arc plasma fabrication of C-band and S-band non-reciprocal ferrite phase shifters.

A powder, to be suitable, has to be deposited at rapid rates and with the required magnetic properties for phase shifter applications. It was estimated that deposit rates required for the economical fabrication of C-band phase shifters would be 30 mil/min/linear in. around a dielectric and 50 mil/min/linear in. for S-band. The required magnetic properties were to be achieved by proper selection of arc plasma spray (APS) parameters and subsequent anneal cycles. A dielectric with a coefficient of thermal expansion, similar to the ferrite sprayed, also had to be established.

INTRODUCTION

The original effort to arc plasma spray ferrites was with nickel zinc ferrite powders.[1] These powders were prepared by flame spray, fluid bed and spray dry processes. APS techniques were developed for spraying thick deposits >20 mil, at deposit rates in excess of 50 mil/min/sq. in. Also, a microwave magnesium manganese ferrite was deposited with good hysteresis properties and low dielectric loss tangent. These results indicated the feasibility of arc plasma spraying a microwave quality ferrite around a dielectric, thus forming the basic ferrite phase shifter configuration.

A ferrite phase shifter is a long ferrite toroid with a dielectric inserted in the center (Figure 1). The length and cross sectional dimensions will vary with different applications and frequencies of operation; however, the fabrication technique remains relatively the same. The current technique used in fabricating this phase shifter requires a ferrite toroid with close dimensional tolerances, machining, and the insertion of a dielectric into the toroid. Some designs require the drawing of cement into the ferrite-dielectric interface to insure that no air voids exist. The assembly of the ferrite and dielectric adds significantly to the cost of the phase shifter and can affect device performance. The arc plasma deposition of a ferrite around a dielectric presents a more economical process for fabricating phase shifters, since an intimate ferrite/dielectric bond can be achieved with the arc plasma process.

The purpose of the arc plasma gun is to melt a ferrite powder and project it onto a target. A schematic of an arc plasma gun is shown in Figure 2. The heat produced by the arc plasma is dependent upon the arc current and arc gas.[2] However, the heat to which the powder is subjected depends on the point of the plasma stream into which the powder is fed.

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1. Richard W. Babbitt, "Arc Plasma Deposition of Nickel Zinc Ferrite", Research and Development Technical Report (ECOM-3597) July 1972, US Army Electronics Technology and Devices Laboratory
2. H. S. Inghem and A. P. Shepard, "Flame Spray Handbook", Vol. III Metco, Inc., Catalog No. 2M999, 1965.

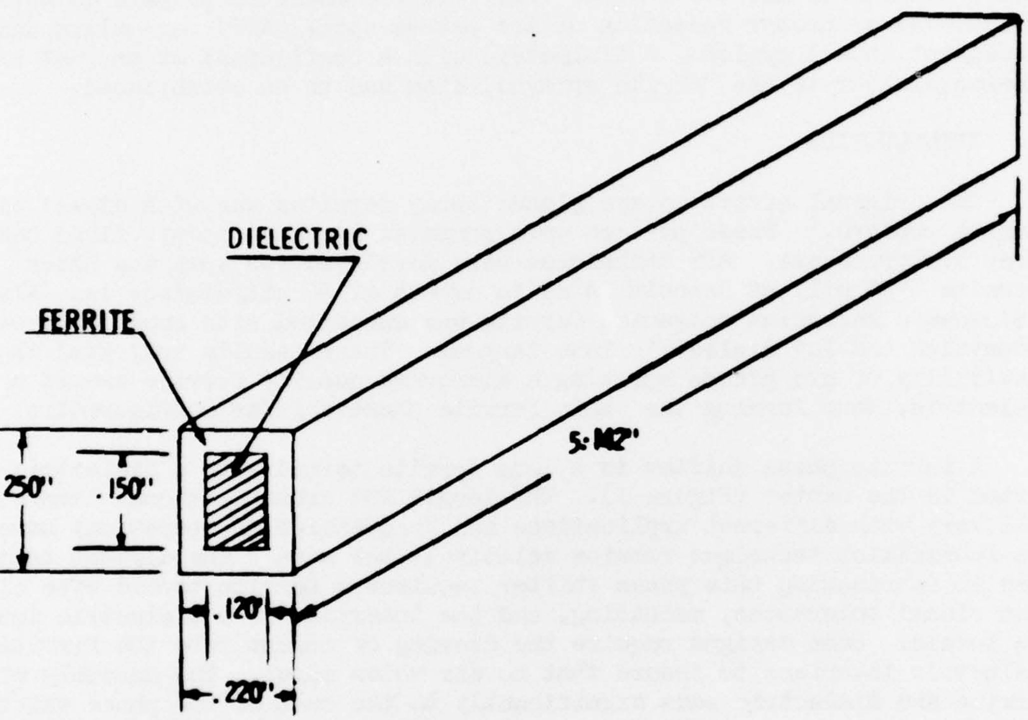

Figure 1. Schematic of a Ferrite Phase Shifter

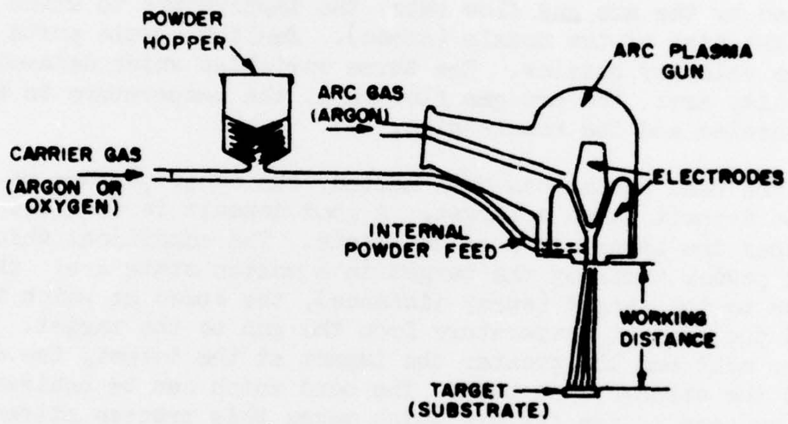

Figure 2. Schematic of Arc Plasma Gun

The powder can be fed internally into the arc gun, or externally at some place in front of the arc gun (Figure 3). The internal feed is the more efficient for melting the powder; however, volatile elements can be lost, oxides are more readily reduced, and "loading" may occur. Loading occurs when the powder melts before leaving the gun (anode), where it resolidifies and builds up, interfering with the plasma stream and results in pieces of the ferrite breaking off and depositing on the target. An external feed eliminates the possibility of loading, but more heat is required to melt the powder causing greater difficulty in feeding all the powder particles into a uniform heat zone of the plasma. A compromise of these two powder feeds is to use a cover (Figure 3) which is not as susceptible to loading as the internal feed,yet restricts the powder to a narrow temperature zone. Regardless of the technique, it is necessary that the powder be fed with velocity capable of penetrating the plasma stream thereby producing a sufficient melt. The velocity of the powder is controlled by the powder carrier gas flow rate and the size of the powder port. After the powder is in the plasma stream, the degree of melt it will experience is dependent upon the heat available and the time in the plasma (dwell time). Dwell time is controlled by the arc gas flow rate, the temperature to which it is heated and the size of the nozzle (anode). Smaller nozzle ports are considered high velocity nozzles. The three variables which determine the plasma velocity are: the arc gas flow rate, the temperature to which the arc gas is heated and the nozzle size.

After the feed powder has been melted, the other purpose of the arc plasma is to deposit it on a target. A good deposit is realized when the powder reaches the target in a molten state. The conditions which contribute to the powder striking the target in a molten state are: the distance from the gun to the target (spray distance), the speed at which the powder travels and the ambient temperature from the gun to the target. The more complete the melt and the greater the impact at the target, the denser the deposit and the stronger the bond. The bond which can be achieved with the arc plasma process is the feature which makes this process attractive for the fabrication of phase shifters. However, for the arc plasma process to be economical, it must deposit ferrite powders at fast rates around a dielectric and with the required magnetic properties.

FERRITE POWDER

a. <u>Powder Characteristic Desired for APS</u> A powder for arc plasma spraying bulk ferrite bodies must be free flowing in order to obtain a uniform and high deposition rate. Two other powder characteristics which influence the spray process are the particle size and size distribution. It has been established that particle size influences the deposit density.[1] Table 1 illustrates the effect of powder particle size on density of arc plasma deposited ferrite.

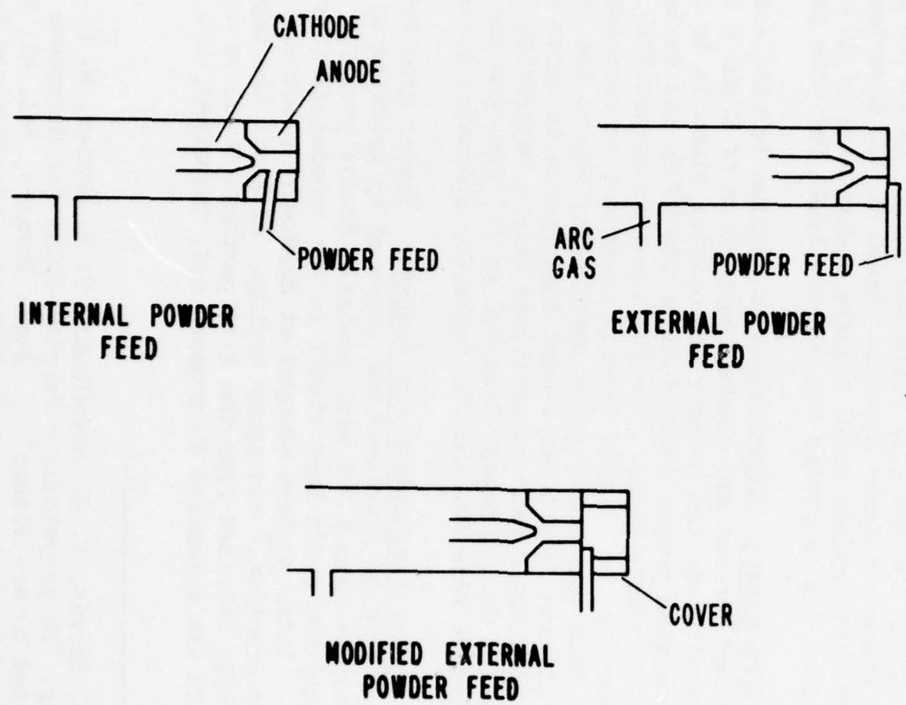

Figure 3. Powder Feed to Arc Plasma Gun

TABLE 1. EFFECTS OF PARTICLE SIZE ON DENSITY

Particle Size (μm)	Arc Current (Amps)	Density (% Theo.)
0.02 - 0.1	350	99%
0.3 - 2.0	500	99%
10.0 - 100	720	98%
10.0 - 100	570	95%

High density deposits were achieved with lower spray parameters (arc current) for small particle sizes. A powder with a large size distribution has a greater tendency to load since the arc plasma parameters are normally set to melt the mean particle size. This causes the smaller particles to melt before clearing the arc gun, while the larger particles are not sufficiently melted to produce a dense deposit. Based on APS experience, an ideal ferrite powder for this program would require a mean size of 10 μm and a 4 to 1 size distribution. Such a powder was not available for this program.

b. Composition Compositions of lithium ferrite and yttrium iron garnet were selected for the arc plasma fabrication of C and S band phase shifters. Lithium ferrite was the primary composition since it is less expensive than garnets and with proper substitutions its $4\pi M_s$ can be tailored for several microwave frequency bands. The yttrium-iron-garnet(YIG) composition was to be an alternate, if lithium ferrite could not be successfully arc plasma sprayed. This was considered a possibility due to the volatility of lithium, and also lithium ferrite at S-band frequencies is known to have high microwave losses. The C-band compositions have a saturation magnetization ($4\pi M_s$) of 1200 gauss for operation from 5.2 to 5.7 GHz, and the $4\ M_s$ for S-band phase shifters were 600 and 750 gauss, for operation between 3 and 4 GHz.

c. Powder Preparation and Analysis Powder used for the original arc plasma spraying of ferrites[3] was prepared by grinding up fully sintered ferrite bodies into a 300 mesh powder. Powder prepared in this manner is expensive and would not lend itself to the economical fabrication of phase shifters. Later powders sprayed at ECOM were processed by flame spraying,[4] fluid-bed reaction,[5] and spray drying. Although the deposits were dense, 99% were readily obtained from the fine particle (0.02 to 0.2 μm) flame spray powder, it was expensive to process and, therefore, not considered for this program.

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3. D. H. Harris, R. J. Janowiecki, C. E. Semler, M. C. Willson, and J. T. Cheng, "Polycrystalline Ferrite Films for Microwave Applications Deposited by Arc Plasma" J. Appl. Physics, Vol. 41, P 1348, 1970.
4. W. W. Malinofsky and R. W. Babbitt, "Fine-Grained Ferrites.I. Nickel Ferrite" J. Appl. Physics, Vol. 32, No. 3, March 1961
5. B. Bavarnick and R. M. Nadkarni "Production Engineering of Ferrite Powder by Fluid Bed Techniques" US Army Electronics Command, Contract DAAB-05-67-C-2708, January 1970.

The fluid bed powder had a small particle size (0.5 to 2.0 μm) and was considered a good powder for this program, but since the manufacturers of microwave ferrites do not have fluid bed equipment available for processing ferrites, it was not selected as the powder process for this program. Microwave ferrite manufacturers have spray drying equipment available to them and, since very fast deposition rates (80 mil/min/sq. in.) had been realized with this powder, it was selected as the ferrite powder type. Ampex's Ferrite Materials Division, because of their willingness and their vast experience with lithium ferrite, was chosen as the supplier of ferrite powders used in this program.

The initial powder evaluated was an Ampex type 3-1202, which was spray dried by two different techniques. Electron microscope photographs of these spray dried powders are shown in Figure 4. Based on these photographs and sieve analysis, it was estimated that spray dried powder "A" had a mean size of 30 microns with a 16:1 size distribution, and powder "B" had a mean size of 50 microns with an 8:1 distribution. The two powders were sprayed with fixed arc plasma spray parameters. Powder A deposited with a 2% higher density than B, but due to its larger size distribution it was more susceptible to loading. Both spray dried powders produced similar hysteresis and device results when arc plasma sprayed. Other spray dried batches of powders were separated into fine, <65 μm, and coarse, >65 μm which, when sprayed, produced significantly different results. The fines were readily deposited with densities from 86% to 96% of theoretical, while the densities of the coarse powders were less than 82% of theoretical. Also, due to the poor melt which occurred with the coarse powder, approximately 600 grams of powder were required to arc plasma spray a 3 in. S-band phase shifter as compared to 200 grams for fine powders. Based on these results, ferrite powder sizes above 75 μm are not acceptable for arc plasma spraying.

Typical powder analysis of spray dried powder as received from Ampex is given in Table 2.

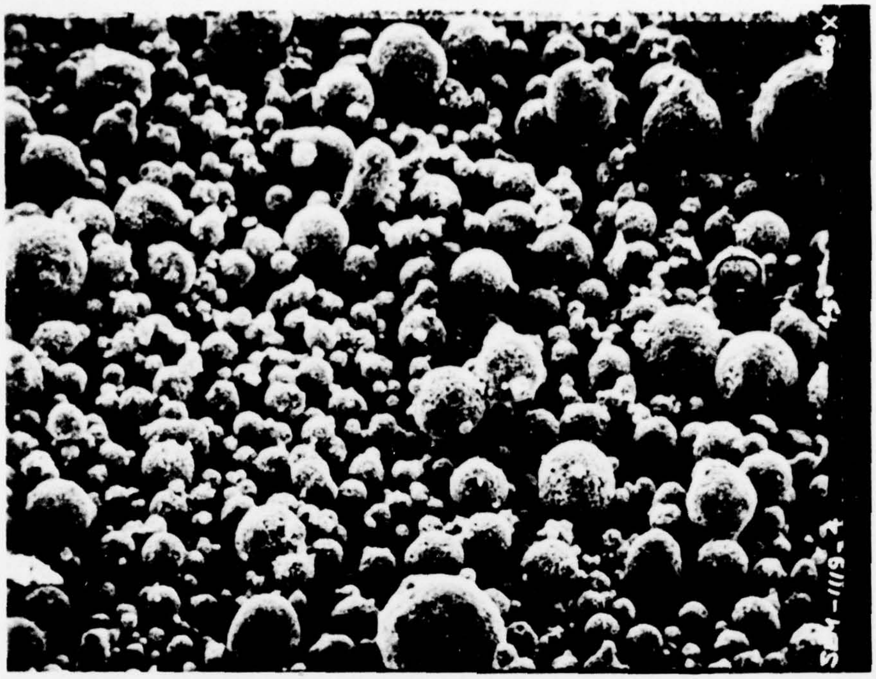
TABLE 2. POWDER ANALYSIS OF SPRAY DRIED LITHIUM FERRITE

Size (μm)	Percent of Powder
>75	21.7
45 to 75	39.3
<45	39.0

This powder is satisfactory as received, but generally to improve device results, the powder particles above 75 μm are removed. There are currently two research and development programs to improve powders for APS. One program is the optimization of spray dry techniques to minimize the coarse powders and the other program is to investigate new powder forming processes.

d. Degree of Reaction The initial Ampex 3-1202 lithium ferrite powder was requested to be a fully reacted composition, which was achieved by precalcining 100°C higher than normally precalcined. (Normally, ferrite powders for conventional sintering are partially reacted in the precalcine phase of preparation). The reason for the fully reacted request was because most of the previous APS work had been done with fully reacted powders. Arc plasma deposited samples of this powder had coercive forces, H_c, which were

A



B

Figure 4. Spray Dried Ferrite Powder

40% greater than reported for standard samples. Even when conventionally sintered, this fully reacted powder had coercive forces 30% higher than realized with partially reacted powder. An arc plasma sprayed sample was delivered to Ampex for evaluation. Ampex noted that in the sprayed sample, bismuth was observed, but after annealing, it was no longer visible. The assumption was made that the high precalcine temperature affected the fluxing action of bismuth, which is added to lithium ferrite compositions to improve density and enhance grain growth[6]. Large grain size is required for low coercive force. Low coercive forces are desired to limit phase shifter drive requirements.

A second lithium ferrite composition, Ampex 3-1200, was evaluated. This composition differed from 3-1202 in that it had a small zinc substitution which reduces coercive force; also it was partially reacted. This powder, when sprayed, did have a lower H_c than sprayed 3-1202 powder, but still higher than its reported value of 0.9 Oe. Also the remanence, B_r, of arc plasma deposited 3-1200 was 650 gauss, significantly lower than the 780 gauss for arc plasma deposited 3-1202. The $4\pi M_s$ of the deposited, partially reacted 3-1200 powder was 1100 gauss as compared to 1200 gauss achieved with the fully reacted 3-1202 powder.

A fully reacted sample of 3-1200 powder was requested in order to resolve if the low $4\pi M_s$'and B_r's were an effect of the zinc substitution or were due to the partially reacted state of the powder. Arc plasma sprayed samples of the fully reacted 3-1200 powder had the same coercive force as the partially reacted samples, but B_r's of 780 gauss and $4\pi M_s$' of 1200 gauss. Figure 5 is the differential phase shift, $\Delta\phi$, as a function of drive current for a fully and partially reacted APS 3-1200 phase shifter. This figure shows the improved device performance and higher phase shift related to spraying fully reacted powder.

e. Deposit Efficiency (Dep. Eff.) and Deposition Rate. Dep.Eff. is the percentage of powder sprayed which is deposited around the dielectric. The Dep. Eff. is very important to the economics of the arc plasma fabrication of phase shifters. Not only does it directly effect the deposition rate, it also effects the amount of powder required to spray a phase shifter. A high Dep. Eff. requires that the ferrite powder be sufficiently melted to adhere to the dielectric and that the powder hit the dielectric. Thus, not only is the aim of the plasma gun important, but also the cross section of the dielectric in relation to the spray span (see Figure 6). Dielectric inserts of most phase shifters are relatively small targets for the arc plasma spray and much of the powder misses the dielectric. The Dep. Eff. for the S-band phase shifter is approximately 40%, as compared to 25% for the smaller C-band dielectric which is sprayed with a cover (see Figure 3) to limit the spray span. Improved cover designs should produce a narrower spray span, increasing Dep. Eff. and deposition rate.

The deposition rate for phase shifter fabrication is defined as the thickness of ferrite deposited in one minute around a one linear inch length of dielectric, (mil/min/in). The deposition rate is dependent on the

6. Paul D. Baba, Gil Argentina, William E. Courtney, Gerald F. Dionne and Donald H. Temme, "Fabrication and Properties of Microwave Lithium Ferrites," IEEE Transaction on Magnetics, Vol. MAG-8, March 1972.

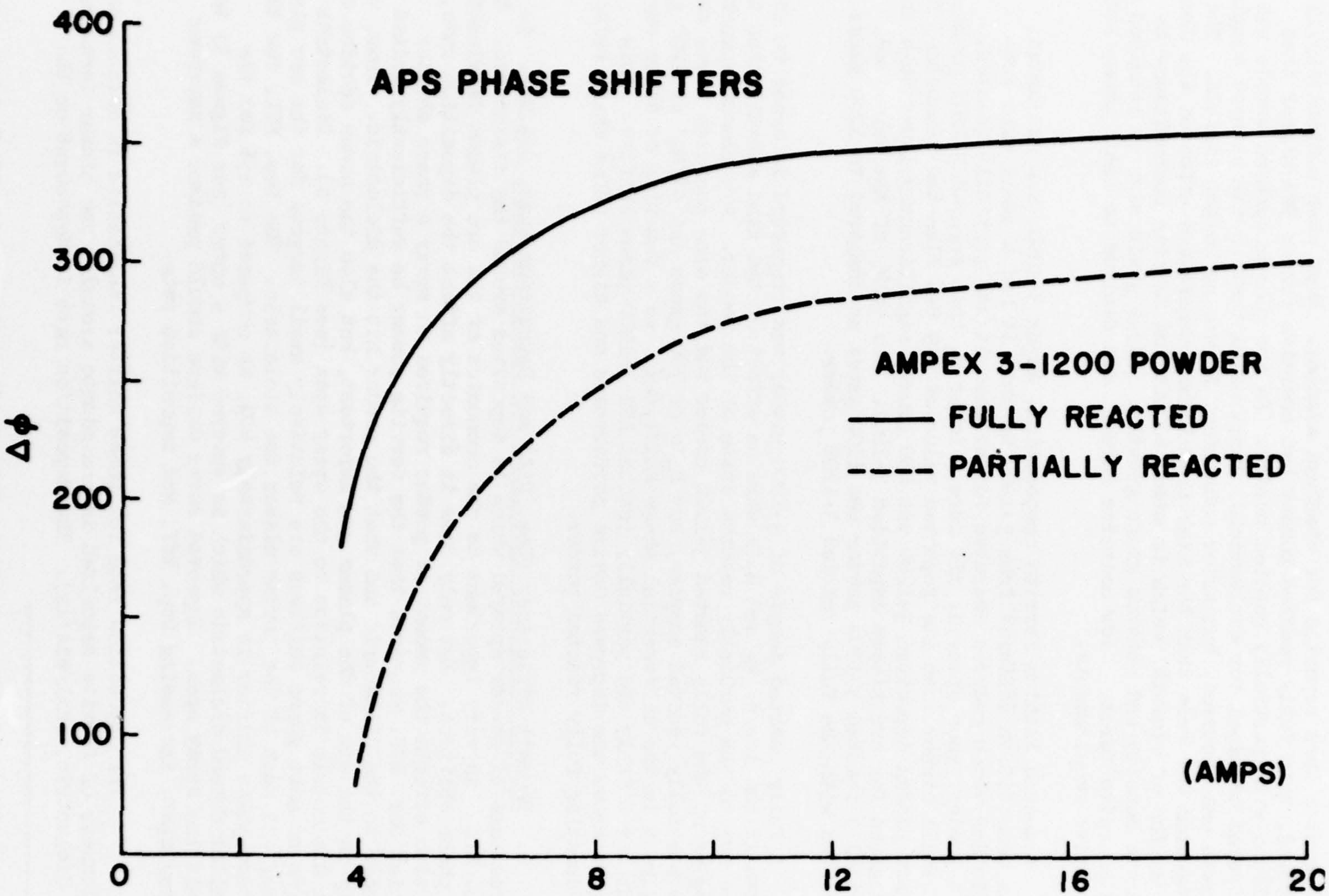


Figure 5. Phase Shifter Results from Fully and Partially Reacted Ferrite Powder

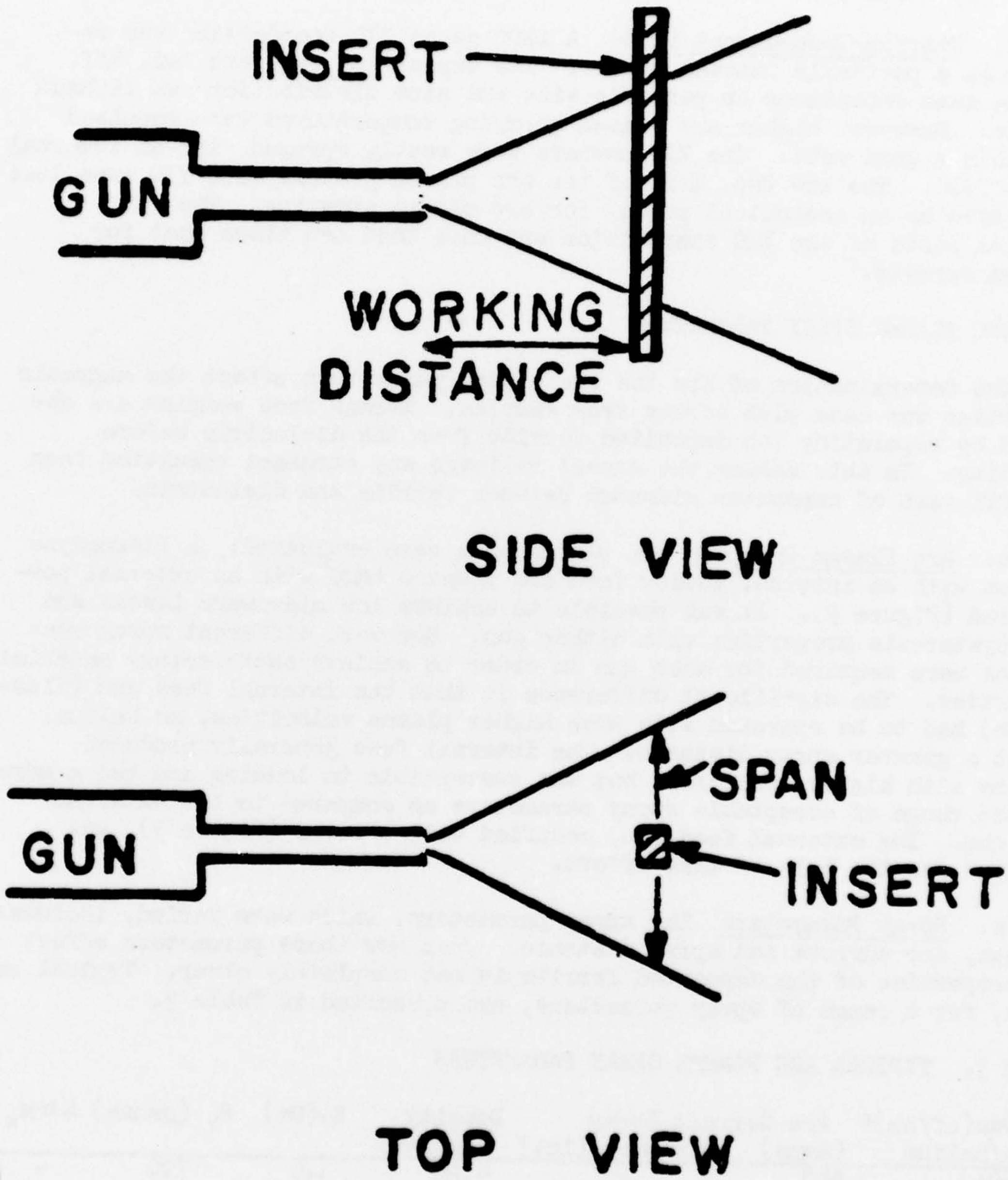


Figure 6. Spray Span from Arc Plasma Gun

Dep. Eff. and the amount of powder fed to the plasma gun. Even with the relatively low Dep. Eff., deposit rates as high as 80 mil/min/in., can be achieved. Normally, 12 grams of powder per minute is fed through the arc plasma gun which produces a deposit rate of 50 mil/min/in.

f. Yttrium-Iron-Garnet (YIG) A 1200 gauss YIG composition was received as a partially reacted powder. Its deposition rate and Def. Eff. had the same dependence on particle size and size distribution as lithium ferrite. However, higher arc plasma spraying temperatures were required to obtain a good melt. The YIG powders were mostly sprayed with an internal powder feed. The low Dep. Eff. of the arc plasma process made YIG even less attractive as an economical powder for arc plasma spraying. The raw material costs of the YIG composition are more than ten times that for lithium ferrite.

ARC PLASMA SPRAY PARAMETERS

The determination of how the arc plasma parameters affect the magnetic properties was done with stress free samples. Stress free samples are obtained by separating the deposited ferrite from the dielectric before annealing. In this manner the anneal relieves any stresses resulting from a coefficient of expansion mismatch between ferrite and dielectric.

a. Arc Plasma Gun Two arc plasma guns were evaluated: a Plasmadyne Minigun with an internal powder feed and a Metco 4MBT with an external powder feed (Figure 3). It was possible to achieve low microwave losses and good hysteresis properties with either gun. However, different spray conditions were required for each gun in order to achieve satisfactory material properties. The significant difference is that the internal feed gun (Plasmadyne) had to be operated with much higher plasma velocities, no helium, and at a greater spray distance. The internal feed generally produced samples with higher densities, but was susceptible to loading and had a more limited range of acceptable spray parameters as compared to the external feed gun. The external feed gun, modified with a cover (Figure 3), was selected for the bulk of this effort.

b. Spray Parameters The spray parameters, which were varied, include: arc gas, arc current and spray distance. Just how these parameters affect the properties of the deposited ferrite is not completely clear. Typical results, for a range of spray parameters, are presented in Table 3.

TABLE 3. TYPICAL ARC PLASMA SPRAY PARAMETERS

Arc Gas(cf/hr)* argon/helium	Arc Current (Amps)	Spray Distance (in.)	Density (gm/cm^2)	H_c(Oe)	B_r (gauss)	$4\pi M_s$
60/5	340	2	4.20	3.0	790	-
70/0	480	2	4.33	2.8	740	-
70/7	400	2	4.18	2.5	760	
70/3	360	2	4.28	3.0	680	1100
100/3	260	1-3/4	3.63	-	-	1100
100/3	300	1-3/4	3.90	2.5	800	1150
100/3	350	1-3/4	4.02	-	-	1145
105/0	580	2	4.28	2.8	580	-
110/3	300	2	4.06	-	-	1210

*cubic feet per hour

Generally, there is good repeatability of results for a given set of parameters when spraying a common powder batch as in Table 4.

TABLE 4. REPEATABILITY OF THE APS PROCESS

Arc Gas(cf/hr) argon/helium	Arc Current (Amps)	Spray Distance (in.)	Density (gm/cm^2)	H_c(Oe)	B_r(gauss)	$4\pi M_s$
70/7	400	2-1/4	4.04	2.6	770	1190
70/7	400	2-1/4	4.18	2.5	760	1180

In practice, the arc current and spray distance is set at some acceptable value, while the arc gas (dwell time) is adjusted to produce a good deposit rate. Two different sets of parameters have produced good C-band phase shifter results. They are:

1. Arc Current - 330 ±10 Amps
   Arc Gas - 75/3 cf/hr
   Spray Distance - 2 ±$\frac{1}{4}$ in.

2. Arc Current - 350 ±10 Amps
   Arc Gas - 40/6 cf/hr
   Spray Distance - 2$\frac{1}{2}$ ±$\frac{1}{4}$ in.

The difference between these two spray conditions is the amount of heat the powder experiences. Condition No. 2, with low argon and high helium, has a longer dwell time and conducts more heat to the powder which are conditions for large particle-size powders.

The S-band phase shifters were sprayed with similar parameters, except the spray distance was greater. This greater spray distance was necessary in order to compensate for the heat build up associated with the wider dielectric.

The arc plasma parameters should be set at a condition which produces a good deposit rate, but not too high a temperature or dwell time, in order to produce a second phase or interaction with the dielectric.

c. <u>Anneal</u> Arc plasma deposited ferrites have high microwave losses and poor hysteresis properties, i.e. high coercive force (H_c) and low remanence (B_r), before annealing. Almost any anneal above 980°C will reduce microwave losses to acceptable values. Table 5 is an example of several anneal cycles and the corresponding dielectric loss tangent (tan δ).

TABLE 5. DIELECTRIC LOSS TANGENT OF ANNEALED ARC PLASMA SPRAYED FERRITE

Sample	Anneal	Tan δ	K'
Commercial	- - - -	1.27×10^{-3}	16.8
APS 1	5 hrs @ 1050°C	0.33×10^{-3}	15.8
APS 2	$\frac{1}{2}$ hr @ 1050°C	0.63×10^{-3}	17.0
APS 3	0 hr @ 1100°C	0.46×10^{-3}	16.8

This table shows that there is not a significant reduction of tan δ between the ½ hour and the 5 hour anneal at 1050°C. However, when very high temperature and long dwell time APS parameters are used, the ferrite may be sufficiently reduced and dense enough to require longer anneal times in order to produce low losses.

Since a range of anneal cycles could produce low loss samples, the main emphasis was to establish an anneal cycle for optimum hysteresis properties. Table 6 shows the hysteresis properties, H_c and B_r, as a function of several anneal cycles.

TABLE 6. EFFECTS OF ANNEAL CYCLE ON HYSTERESIS PROPERTIES OF ARC PLASMA DEPOSITED LITHIUM FERRITE

Anneal Temp(°C)	Zero Soak Time H_c(Oe)	Zero Soak Time B_r(G)	1 Hour Soak Time H_c(Oe)	1 Hour Soak Time B_r(G)	4 Hours Soak Time H_c(Oe)	4 Hours Soak Time B_r(G)
950	4.2	505	3.6	795	3.2	680
990	3.65	790	3.3	785	2.5	730
1030	3.8	810	2.8	730	2.4	610
1070	2.9	790	1.6	630	1.8	460
1130	1.3	675	1.25	740	1.75	295

The anneal temperature ranged from 950°C to 1130°C with soak times from zero to 4 hours. Except for the lowest temperature, 950°C, B_r was maximum for minimum soak time, while H_c would make a significant drop from zero and 1 hour soak and then decrease more gradually. Based on these results, it was concluded that exposure of the sample at higher temperatures and shorter soak times produced the lower H_c with highest B_r values. The anneal cycle established for the C-band shifter was from ¼ to 1 hour at 1030°C, and for S-band 1½ hours at 980°C. The longer soak time established for S-band is attributed to the longer oxygen diffusion time required for the thicker ferrite deposit.

d. Yttrium Iron Garnet (YIG) Yttrium iron garnet could be readily arc plasma sprayed with either the internal or external powder feed gun, since YIG composition is not as volatile as lithium ferrite. However, in order to optimize the hysteresis properties, anneal temperatures in excess of 1300°C for more than 5 hours were required. These anneal conditions resulted in an interaction between YIG and its matching dielectric insert, zinc titanate, when fabricating phase shifters. Thus, at this point further effort with the rare earth compositions was discontinued.

e. Ferrite-Dielectric Match Suitable arc plasma spray parameters and anneal cycles having been established, it was still not possible to fabricate a good phase shifter by arc plasma spraying. All the preliminary evaluation was done on stress free samples. However, when the ferrite was annealed while still bonded to the dielectric, stresses were generated from the coefficient of expansion mismatch between ferrite and dielectric which degraded the hysteresis properties.[7] The significance of this degradation

7. E. Stern and D. Temme, "Magnetostriction Effects in Remanence Phase Shifters," IEEE Trans., Vol. Mtt-13, p 873, Nov 1965

is demonstrated by the hysteresis results of a 1202 powder with a coefficient of linear expansion of $14.1 \times 10^{-6}/^{o}C$ deposited and annealed around a lithium titanate dielectric with a coefficient of $19.0 \times 10^{-6}/^{o}C$. A cross sectional slice of the phase shifter element was cut into two concentric toroids, as depicted in Figure 7. The inner toroid, nearest the dielectric, would exhibit the maximum stress effect. The hysteresis loops in Figure 7 show the effects of the stresses generated by the coefficient of expansion mismatch. Remanence was lower and coercive force higher in the highly stressed inner toroid when compared to the outer toroid. In order to confirm that the degradation of the hysteresis properties was due to stresses and not to an interaction with the dielectric, both toroids were reannealed to relax stresses, i.e. to produce stress-free toroids. After the anneal the hysteresis loop of the inner toroid improved considerably as illustrated in Figure 7. The outer toroid had only a small improvement which indicates that some stresses were originally present in this toroid, i.e.(at this distance from the dielectric). This degradation was found to be present with either a compressive or a tensile stress. Since a 50 mil ferrite wall thickness was required for the C-band phase shifter element, dielectrics with coefficients of thermal expansion similar to lithium ferrite had to be developed.

DIELECTRICS FOR APS PHASE SHIFTERS

A program to develop dielectrics for inserts for the APS fabricated lithium and yttrium iron garnet phase shifters was initiated. The dielectric requirements were: a) low microwave loss, b) high dielectric constant(K), and c) a similar thermal coefficient of linear expansion as the ferrite to be deposited. R. J. Brandmayr of the ceramic group of the US Army Electronics Technology and Devices Laboratory was responsible for this effort.

a. Dielectric Development (by R. J. Brandmayr) In the manufacture of ferrite phase shifters it becomes necessary to bond the ferrite toroid which can be a YIG ferrite or a lithium ferrite to a low loss ceramic dielectric substrate by use of an epoxy or similar type resin. While this technique works to some extent, it also has serious problem areas such as air entrapment, improper alignment of the dielectric insert, and high costs. Hence, the arc plasma process was devised as a potential means to fabricate these phase shifters by spraying ferrite powders through the arc plasma onto a suitable dielectric substrate, thus eliminating the problems associated with cementing the pieces together. However, a significant impediment to the success of the program existed in trying to obtain suitable substrates having the desired compatibility with ferrites and also the required microwave properties of high K, low loss and low TCK. The Frequency Control and Signal Processing Devices Technical Area of the Electronics Command has been actively engaged in development of microwave dielectrics having these properties, and a number of materials have been developed having properties close to those desired for this program.[8] The properties of the dielectric were required to match YIG and lithium ferrites shown in Table 7. Zinc titanate dielectric (ZT-75) which was developed under an ECOM contract

8. D. W. Readey, C. P. Hastwig, E. A. Maguire, D. J. Masse', "Microwave High Dielectric Constant Materials," Technical Report ECOM-0455-F, June 1971.

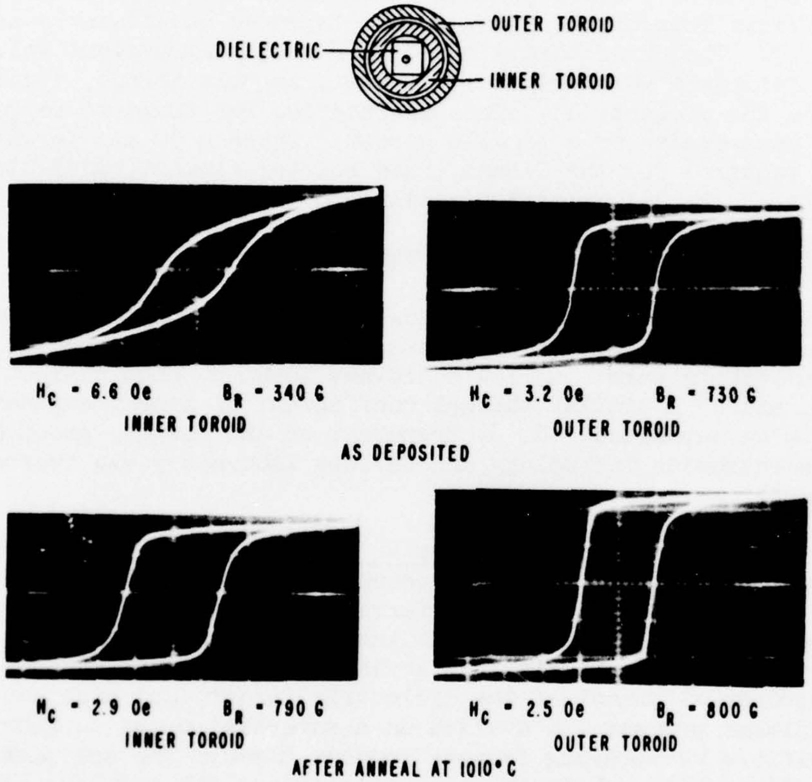


Figure 7. Effects of Stress from Coefficient of Expansion Mismatch

was determined to have the necessary coefficient of thermal expansion properties and microwave electrical properties and serves as a good substrate for YIG ferrite toroids.

TABLE 7. DESIRED PROPERTIES OF DIELECTRICS FOR YIG AND LITHIUM FERRITE

PROPERTY	YIG SUBSTRATE	LITHIUM FERRITE SUBSTRATE
Coefficient of Thermal Expansion	11.5×10^{-6}	15×10^{-6}
Maximum Safe Operating Temperature and Time	1400°C for 4 hours	1000°C for 4 hours
K (1-9 GHz)	30	30
TAN δ	0.0005	0.0005
TCK	-400 PPM/°C	-400 PPM/°C

It was decided that ZT-75 should be modified by adjusting the TiO_2 content to attempt to improve the dielectric constant without seriously affecting the other properties. This material was found to be a successful substrate for YIG ferrite toroids. Bars of modified ZT-75 of dimensions 6 in. x 3/4 in. x ½ in. were purchased from the Raytheon Company. The properties of these bars are shown in Table 8.

TABLE 8. PROPERTIES OF MODIFIED MICROWAVE DIELECTRICS[9]

	LITHIUM TITANATE	ZINC TITANATE
X-band Dielectric Constant	24.42	25.21
Temperature Coefficient of Dielectric Constant	-10.0	-81.7 ppm/°C
X-band Dielectric Loss Tangent	2.64×10^{-4}	8.46×10^{-4}
Coefficient of Thermal Expansion ($\times 10^{-6}$/°C)	15.5, 18.59	11.20

A lithium titanate body Li_2O - 1.5 TiO_2 which was also developed under an ECOM contract, was recommended for use as a substrate for arc plasma sprayed lithium ferrite toroids. However, it was decided to try to lower the coefficient of thermal expansion of this body to about 15×10^{-6} from 19×10^{-6} so as to match that of lithium ferrite. Samples 6 in. x 3/4 in. x 1½ in., were also purchased from Raytheon and the properties of this material are given in Table 9.

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9. E. A. Maguire and D. W. Readey, "Some Microwave Dielectric Materials in the LiO_2 - TiO_2 - Al_2O_3 System," ACS Electronics Div., September 1974.

TABLE 9. MATCHING FERRITE AND DIELECTRIC COMBINATIONS

Ampex Ferrite Type	$4\pi M_s$	Dielectric	α	K
3-601 (fully reacted)	600	Ampex Type 244-9-1 Lithium Titanate	17.6	27
3-750 (fully reacted)	750	Ampex Type 244-9-1 Lithium Titanate	17.6	27
3-1200 (partially reacted)	1200	Ampex Type 222-2A Lithium Titanate	13.1	28
3-1200 (fully reacted)	1200	Non-Mag Lithium Ferrite Type 180(3)	14.6	19.5
3-1202 (fully reacted)	1200	Non-Mag Lithium Ferrite Type 190(3)	14.9	20.0

The samples were processed by weighing out into titanium mills. Spheres of titanium were also used as the grinding media and methyl alcohol was used as the liquid medium. The batches were smelted for about 16 hours and the mills were emptied into open pans which were dried at about 60°C. The drier material was screened and granulated through a sieve and calcined in zirconia crucibles for ten hours in air at 900°C. After calcining, the batches were milled and granulated again. The samples were isostatically pressed and fired in a kiln. The firing temperatures were 1260°C for both lithium titanate and ZT-75. For this work the lithium titanate was modified with alumina additions to lower the coefficient of thermal expansion and the ZT-75 was modified with additional titanate.

Trans-Tech Inc., of Gaithersburg, Md., was also contacted for the purpose of purchasing modified samples of their D-30 body in an attempt to increase the coefficient of thermal expansion, of this body, to 11.5×10^{-6} and/or 15×10^{-6} so as to match the YIG and lithium ferrites. A purchase request was processed in order to acquire samples of D-30 body which was modified with magnesium additions.

b. Dielectric Ferrite Match In addition to the zinc titanate and lithium titanate dielectrics developed, a series of non-magnetic lithium ferrites, developed by Dr. Van Hook at Raytheon, were also evaluated for compatibility with arc plasma sprayed ferrites.

The zinc titanate dielectric had a good coefficient of expansion match with yttrium iron garnet. However, the zinc titanate interacted with the garnet during the required high temperature anneals (above 1300°C).

Several suitable combinations of lithium ferrite and dielectric have been established. Table 9 gives the most favorable ferrite-dielectric combinations, i.e. those which produce the best hysteresis properties. It is interesting to note that the fully and partially reacted 3-1200 powders required different dielectrics for optimum hysteresis properties.

ARC PLASMA FABRICATED PHASE SHIFTERS

a. Fabrication Technique Using established spray parameters with an appropriate anneal cycle, it is possible to arc plasma spray lithium ferrite, with suitable microwave properties, around a dielectric which has

a close matching coefficient of expansion. The arc plasma fabrication process is done by spraying ferrite on a rotating dielectric. This spraying is done in an oven (Figure 8) which is preheated to decrease thermal shock and enhance ferrite deposition. While the dielectric is rotating, it is pulled passed the stationary arc plasma gun once, until the desired length is coated. The rate of pull and the ferrite deposition rate determine the deposit thickness. The ferrite is over-sprayed from 20 to 50 mil, and then the outer dimensions are machined to tolerance. Figure 9 shows a C-band phase shifter as deposited and after machining. The dielectric, which extends beyond the deposited ferrite, is used for a machining reference.

Utilizing this fabrication technique, no expensive tooling is required for design changes of the phase shifter, or for spraying phase shifters for different frequency bands. Figure 10 shows two differently designed C-band phase shifters which were sprayed in sequence. This only required that a different size dielectric be used and the rate of pull adjusted for the change in ferrite wall thickness.

There are some areas in this fabrication technique where it becomes necessary to exercise care. For example, the gun must not be too hot or be too close to the dielectric to cause an irreversible chemical phase change or a strong ferrite dielectric interaction. Also, the rotation speed of the dielectric must be fast enough to avoid thermal cracks in the ferrite. Thermal cracks can occur during slow rotation, since there is time for sufficient cooling when one side of the target (ferrite/dielectric) is turned away from the arc gun, which reheats when turned toward the gun. This heating and cooling effect has caused cracks in the ferrite deposit. Though no safe rotation speed has been established, speeds above 60 rpm are considered satisfactory.

b. Fabrication Time The fabrication time is dependent on ferrite deposition rate and phase shifter dimensions. The economics of the arc plasma process is dependent upon a high deposition rate. Initially, for the C-band phase shifter (Figure 9) it was estimated that a minimum deposit rate of 30 mil/min/linear inch would be necessary for the APS process to be considered economical. Deposition rates of lithium ferrite around dielectrics have been increased from 20 to 80 mil/min/linear inch during this two year effort. This increased deposit rate is attributed to improved powders, powder feed procedures and better spraying techniques.

Figure 11 shows the cross sections of several arc plasma fabricated phase shifters. Based on deposition rates from 30 to 80 mil/min/linear inch, Table 10 gives the fabrication time for each of these phase shifters. Allowing approximately one dollar per minute spraying cost, these fabrication rates show that for higher frequency phase shifters, the arc plasma process is most economical.

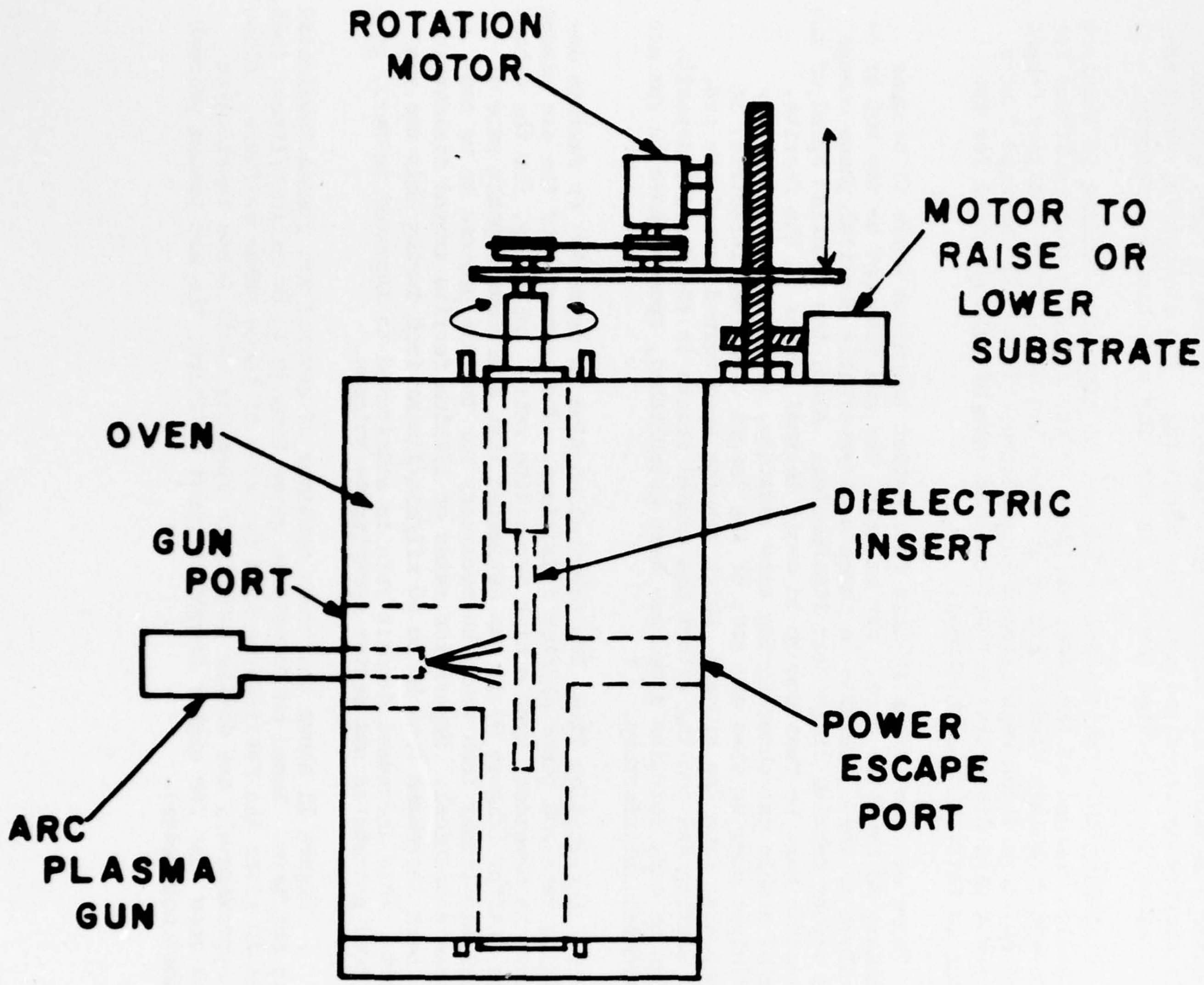

Figure 8. Oven and Equipment for Spraying Ferrite Around Dielectric Insert

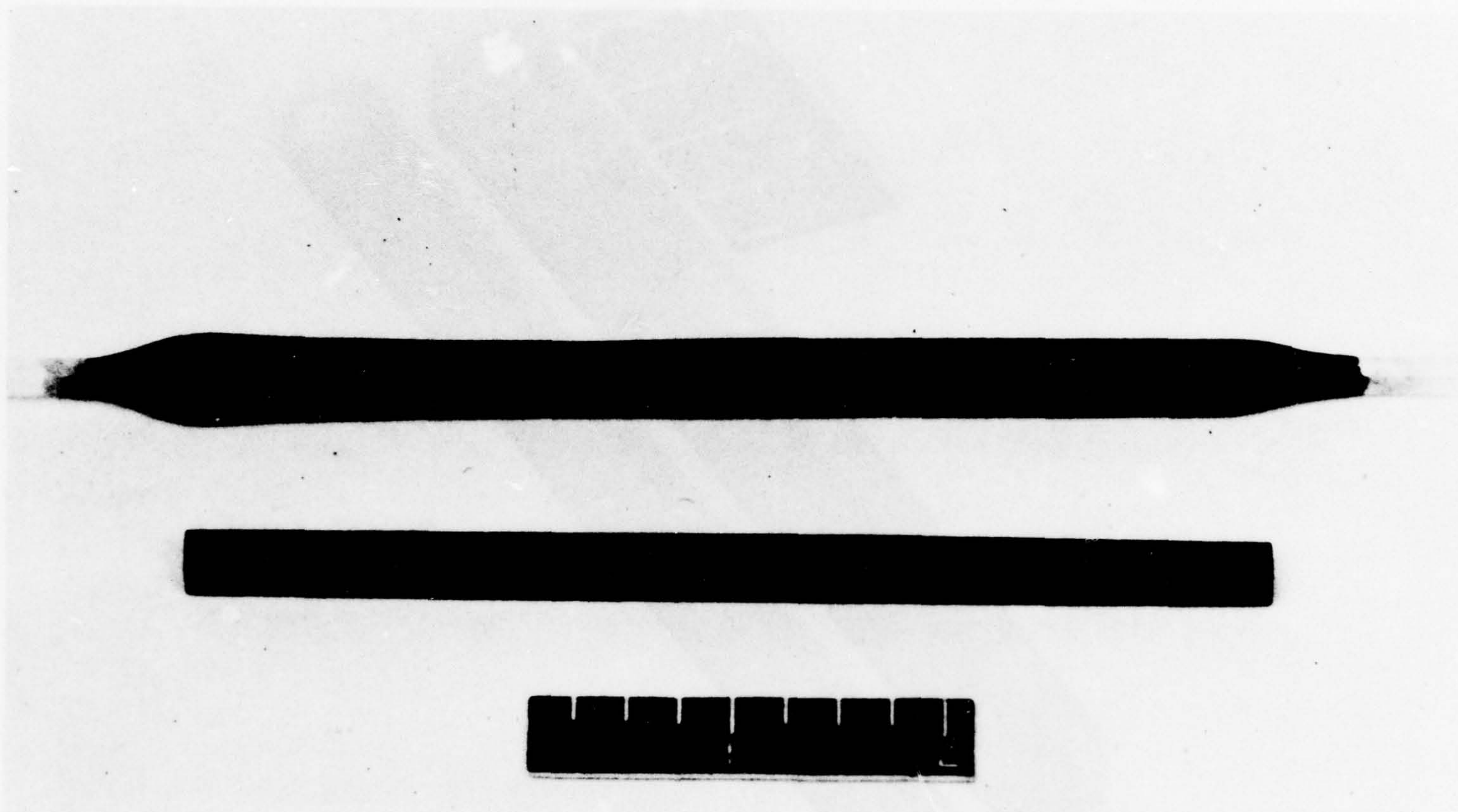

Figure 9. Arc Plasma Ferrite Phase Shifter, as Deposited and after Machining

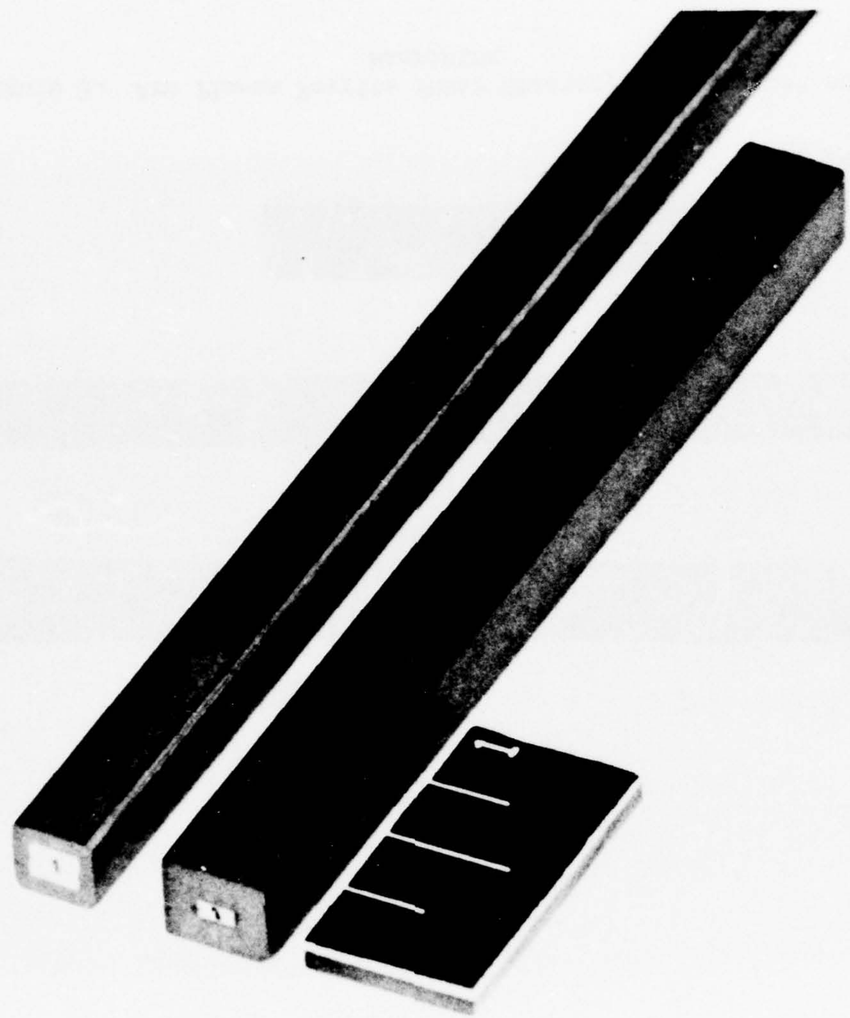

Figure 10. Variations of APS C-band Phase Shifters

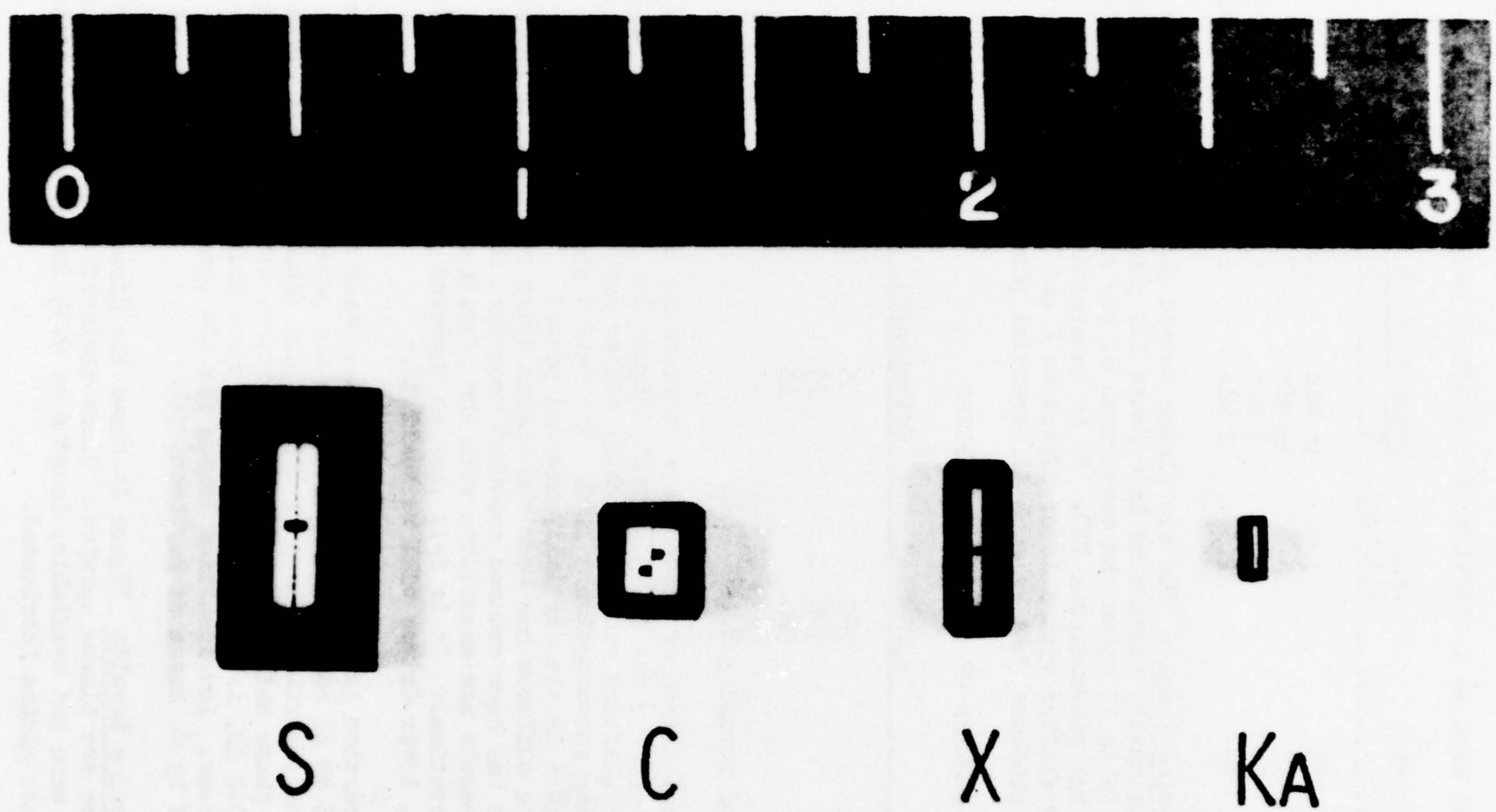

Figure 11. Cross-Section of Arc Plasma Sprayed Phase Shifters

TABLE 10. SPRAYING TIME AS A FUNCTION OF DEPOSITION RATE

Frequency	Ferrite Thickness (Mil)	Length (inches)	Deposition Rate (mil/min/in) 30	50	80
S	125	7.0	35 min	21 min	13 min
C	50	5.1	13 min	7.5 min	5 min
X	55	2.5	7 min	4.5 min	2.5 min
Ka	20	1.5	2 min	1.2 min	0.75 min

c. C-band Device Results The arc plasma C-band phase shifters were compared to conventionally fabricated 1200 Gauss YIG phase shifters of the same dimensions. Table 11 shows the comparison of the APS lithium ferrite phase shifters to the conventional YIG. It is interesting to note that the APS 1202 phase shifter with a lower effective K and lower B_r than the conventional YIG, produces just as much differential phase shift.

TABLE 11. DEVICE RESULTS OF APS PHASE SHIFTERS

Sample	B_r (Gauss)	Drive(Amps)	$\Delta\phi$	Loss(dB)
Conventional YIG	780	6	350°	0.9
APS 1202	700	15	350°	0.9
APS 1200	560	15	300°	0.7

Length of toroid = 5.14"

A more useful comparison of these phase shifters is the plot of differential phase shift ($\Delta\phi$) vs. drive current, Figure 12. These curves show that the APS phase shifters require slightly higher drive currents which can be reduced with zinc substitution 3-1200. The 3-1200 phase shifter depicted in this figure is the partially reacted powder. The main shortcoming of APS phase shifters has been the higher drive requirement, though more recently this has been reduced somewhat from that of Figure 12. The high drive requirements are associated with the ferrite density which is less than 95% theoretical. It is felt that as improved powders for spraying are developed, lower drives will be realized.

Recent insertion losses of the APS C-band phase shifters are generally less than 0.5 dB as compared to 0.9 dB for the conventional YIG. Figure 13 shows the insertional loss (1 dB/Div) and return loss (10 dB/Div) for an APS C-band phase shifter. This phase shifter, with an insertion loss of approximately 0.2 dB, is one of several APS phase shifters which exhibit these very low losses. Low insertion losses for APS phase shifters have also been measured by D. Masse at Raytheon.

d. S-band Device Results Figure 14 shows the dimensions of the S-band phase shifter to be arc plasma sprayed. Since dielectrics long enough for the 7 inch length were not available, lengths of 2.33 inches and 3.5 inches (Figure 15) were arc plasma fabricated.

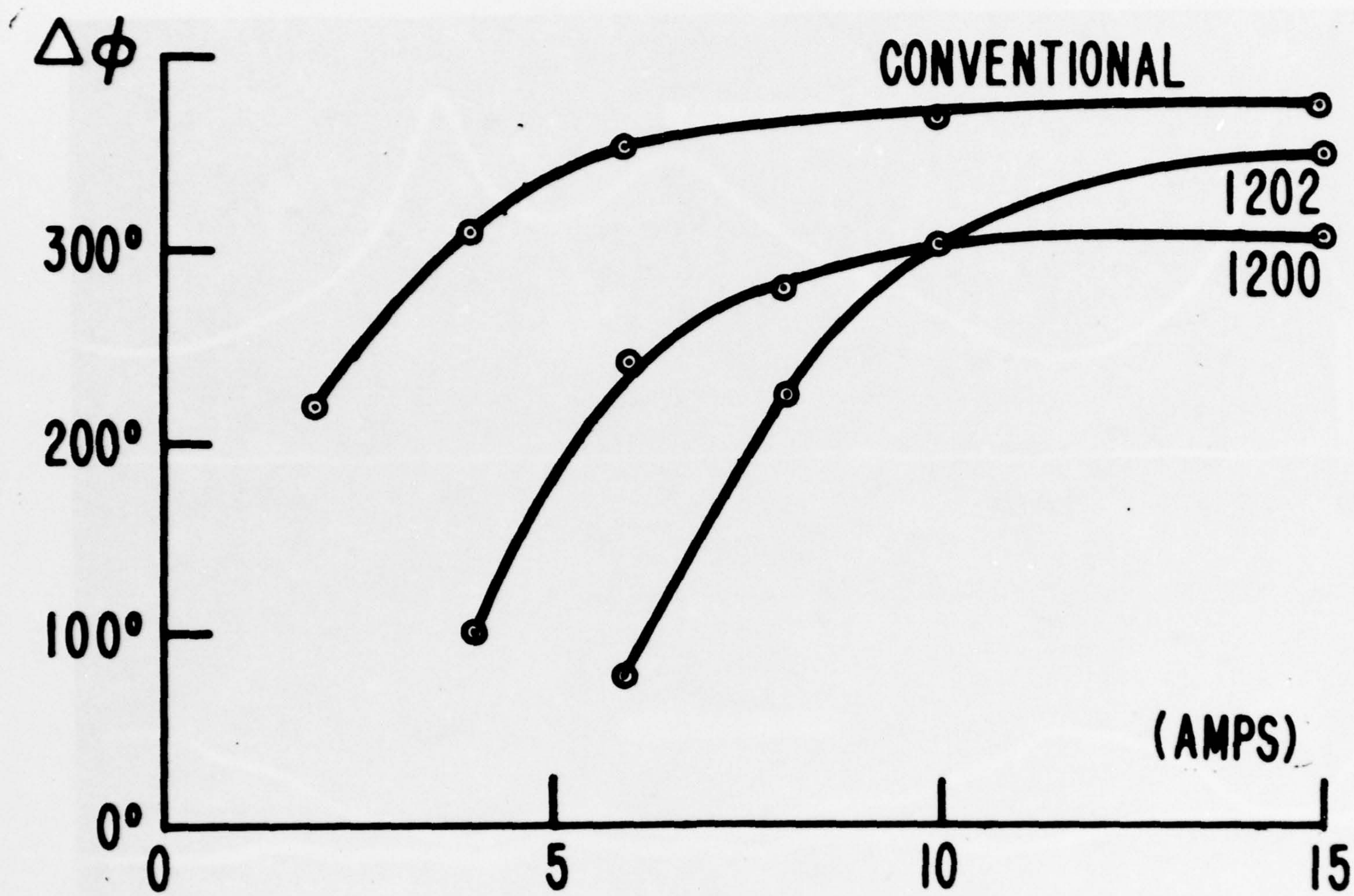

Figure 12. Comparison of C-band APS to Conventional Phase Shifters

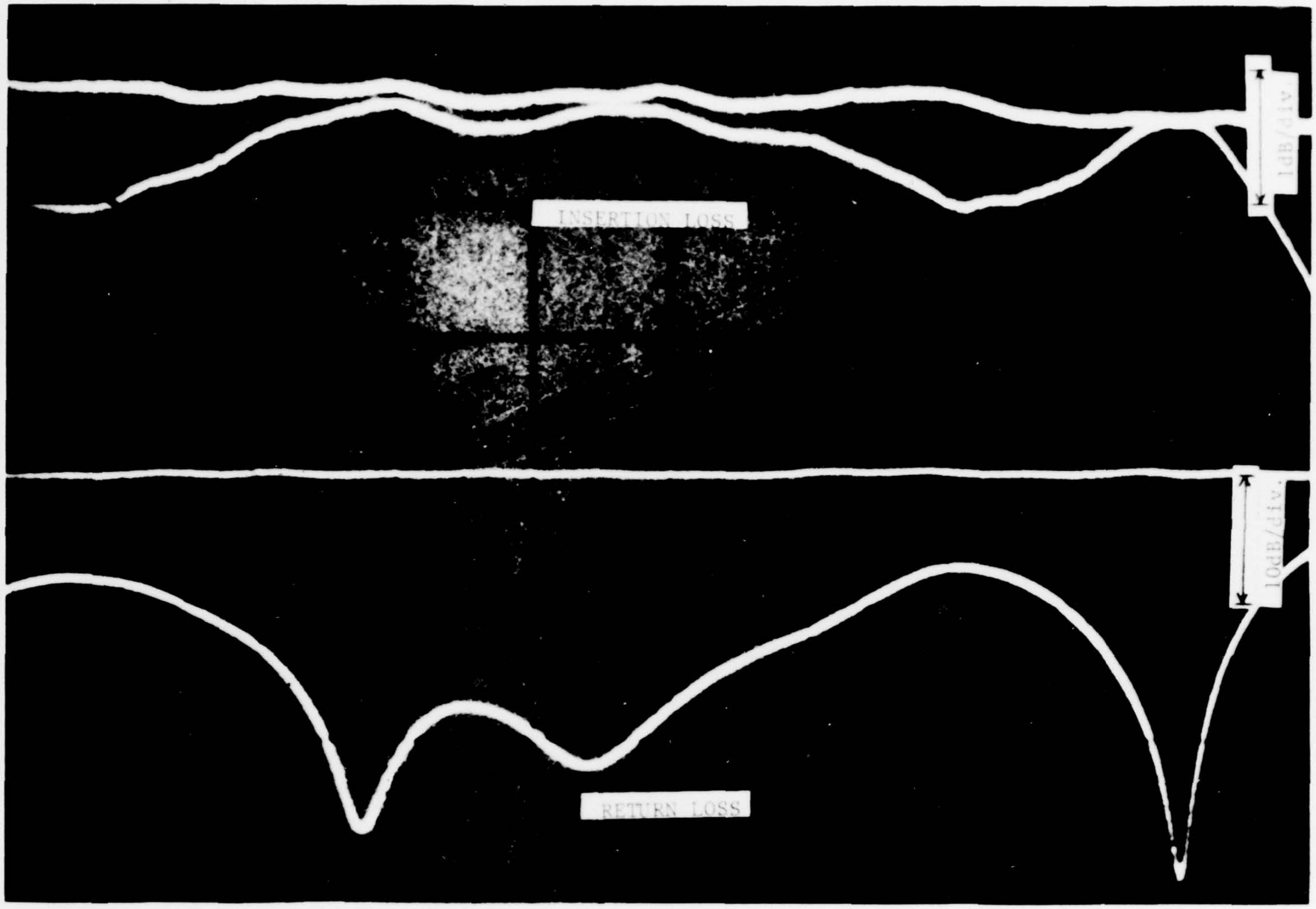

Figure 13. Insertion Loss and Return Loss for APS C-Band Phase Shifter

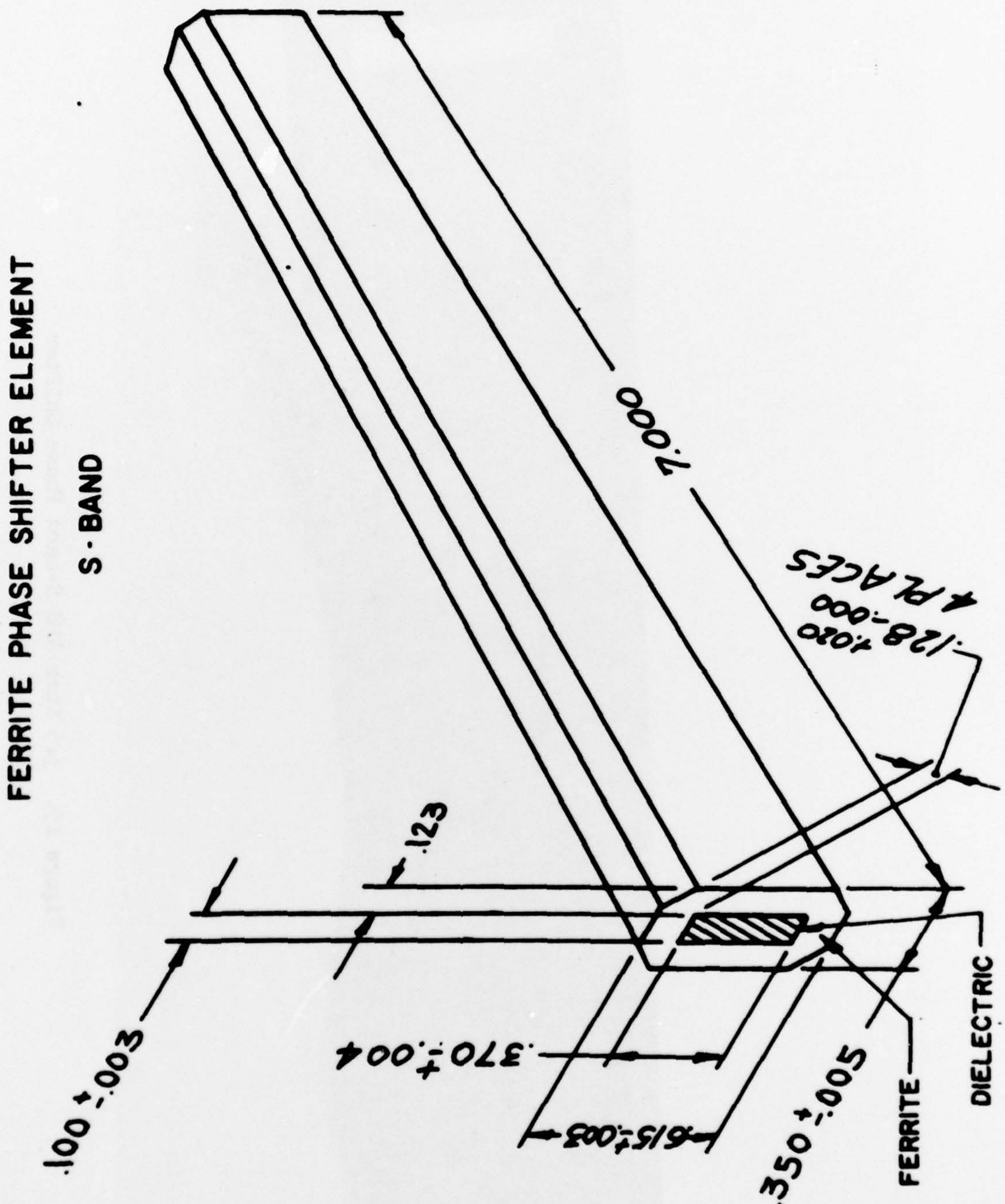

Figure 14. Dimensions of S-band Phase Shifter

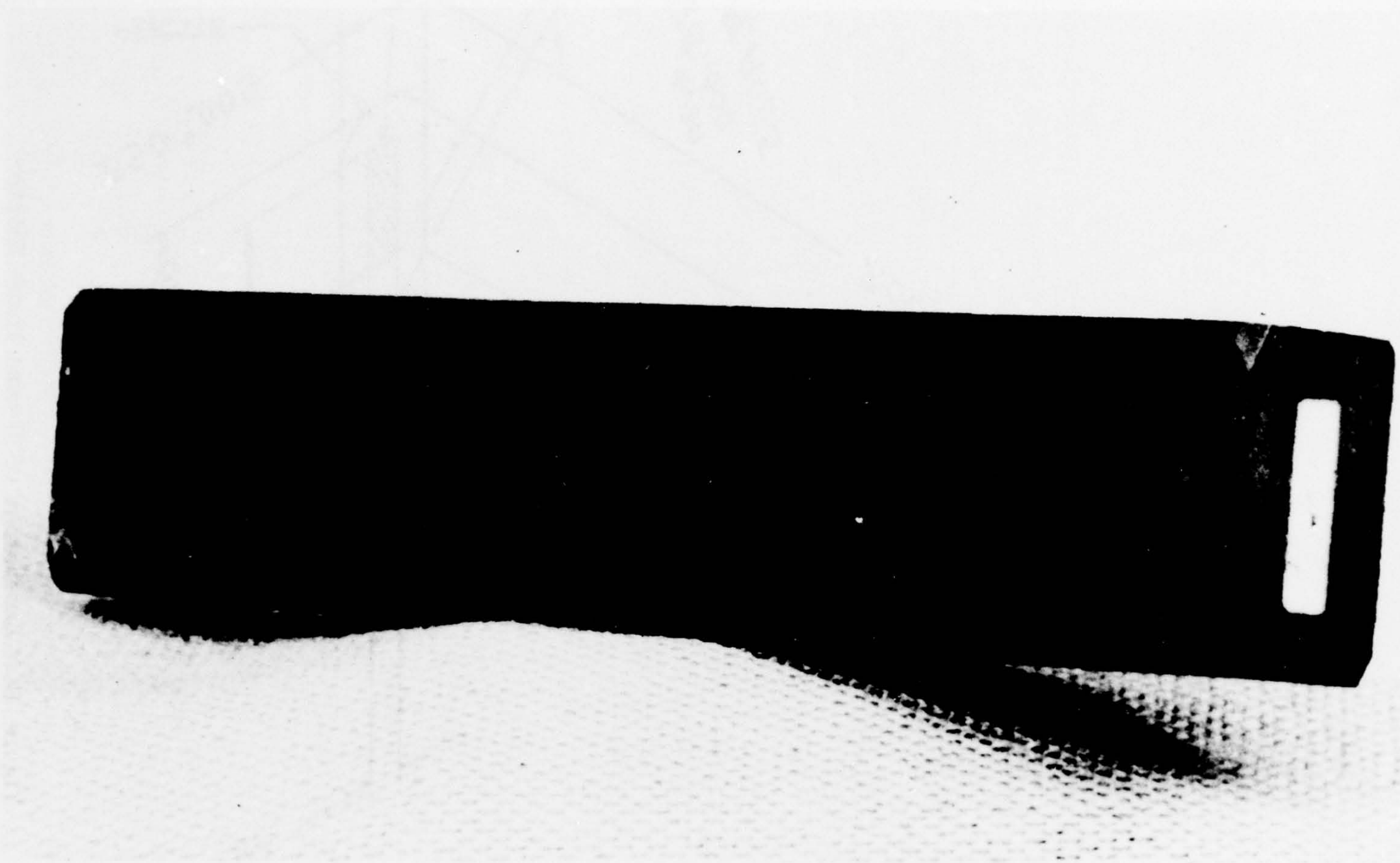

Figure 15. 3.5 inch APS S-band Phase Shifter

Two S-band lithium ferrite compositions were evaluated: an Ampex 600 Gauss composition with a cobalt substitution and a 750 Gauss composition. The ΔH_k of these compositions when arc plasma sprayed were 8.85 Oe and 3.0 Oe, respectively. These values are 50% greater than reported for the same composition conventionally sintered. This increase of ΔH_k, which is a measure of power capacity, is attributed to reduced grain size. However, this increased ΔH_k, coupled with lower magnetic loss, μ'', results in higher $\Delta H_k/\Delta H_{eff}$ ratios, which is important for certain power applications. The ratio for arc plasma S-band lithium ferrites is 0.35 as compared to 0.19 for conventionally sintered samples. This is a significant improvement. Table 12 summarizes these material improvements for arc plasma sprayed 3-601 and 3-750, and compares these results to a conventional YIG composition and a conventional lithium ferrite.

TABLE 12. MAGNETIC LOSS AND ΔH_k OF APS LITHIUM FERRITE

Sample	ΔH_k (Oe)	u"	ΔH_{eff}(Oe)	$\Delta H_k/\Delta H_{eff}$
APS 3-750	2.96	0.0031	9.49	0.312
APS 3-601	8.85	0.0061	23.32	0.379
*Conventional YIG	5.97	0.0033	11.55	0.519
**Conventional Li Ferrite 3-755	5.05	0.0084	25.50	0.198

*Trans-Tech G-600
**Ampex 3-755

Arc plasma sprayed 600 Gauss phase shifter required the full 7" for 360° differential phase shift, while the 750 Gauss composition required only 4.5" lengths. Like the C-band arc plasma phase shifters, the S-band was compared to a 680 Gauss gadolinium doped yttrium iron garnet. Figure 16 is the comparison of the APS 601 and APS 750 to the standard YIG. The remanences of the APS 601 and 750 are 250 gauss and 380 gauss respectively. The two 3-601 curves represent two different dielectric inserts, one of which produced more stresses, thereby reducing the amount of differential phase shift. The insertion loss of a 7" phase shifter, two 3.5" sections, is 1.0 dB. These values of remanence and loss are not felt to be optimum since not as much effort has been spent on S-band as C-band and the dielectric is not matched as well.

e. Temperature Dependence and Magnetostriction The high drive currents, i.e. high coercive forces, made it difficult to achieve reliable temperature dependence measurements on the arc plasma phase shifters. With the drive current available, 15 Amps, and with the hysteresis measurement equipment, it was not always possible to be on a major hysteresis loop. Figure 17 shows how B_r varies with temperature from -30° to +80°C. The drive currents for the APS samples were approximately 4 times the coercive force,

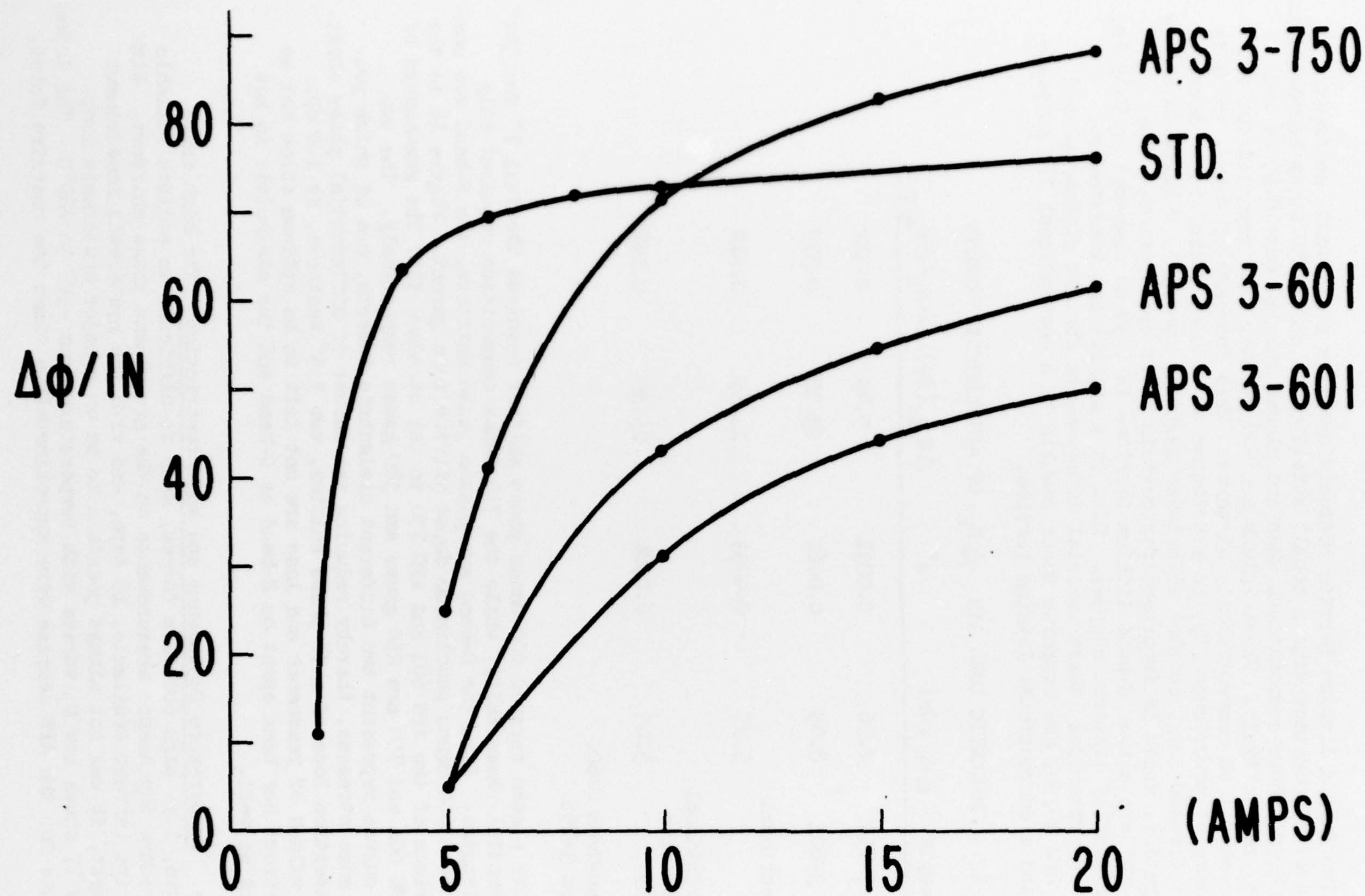

Figure 16. Performance of APS and Conventional S-band Phase Shifters

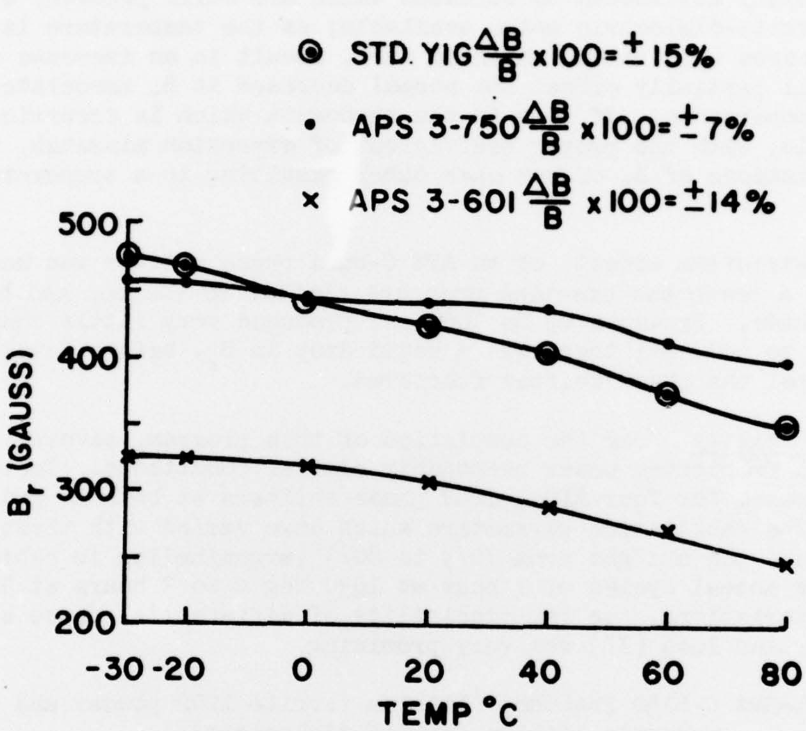

Figure 17. Temperature Dependence of APS Phase Shifters

and it is felt that the values of $\pm 7\%$ and $\pm 14\%$ variation in B_r over the specified temperature range are fairly reliable. This temperature stability is as good or better than that for the standard YIG. A zinc substituted APS 3-1200 phase shifter was driven in excess of 5 times the coercive force and yielded a B_r variation of $\pm 10\%$ from $-30^\circ C$ to $+80^\circ C$. This is better than a conventionally sintered sample and as good as the standard YIG composition selected for phased array applications. This improvement in temperature dependence is primarily attributed to stresses which are still present, even with the best ferrite-dielectric match available; as the temperature is increased, the stresses will relax, which in turn, result in an increase of B_r. This increase will partially offset the normal decrease in B_r associated with a rise in temperature. If this is the phenomena which is occurring, it it may be possible, with the proper coefficient of expansion mismatch, to have the two variations of B_r offset each other resulting in a temperature stable B_r.

The magnetostrictive effect of an APS C-band phase shifter was measured by placing it in a press and exerting pressure similar to the top and bottom walls of a waveguide. Pressure up to 3000 psi produced very little change in B_r; from 3000 to 5000 psi there was a rapid drop in B_r, before leveling off. At 10,000 psi the phase shifter fractured.

f. Reproducibility Near the completion of this program, several phase shifters had been fabricated under reasonably similar conditions. Table 13 shows the performance for four APS 3-1202 phase shifters at both 10 and 15 ampere drives. The fabrication parameters which were varied with these four phase shifters were the arc gas from 70/3 to 80/3 (argon/helium in cubic feet per hour) and the anneal cycles of 1 hour at 1030 deg C to 3 hours at $960^\circ C$. Even with these variations, the reproducibility of differential phase shift ($\Delta\phi$) and insertion loss (IL) was very promising.

TABLE 13. ARC PLASMA C-BAND PHASORS; (lithium ferrite 1202 powder and non-magnetic lithium ferrite dielectric).

Phasor No.	Drive Current Amperes	$\Delta\phi$	IL (dB)	B_r (Gauss)	H_c (Oe)
1	10	300°	0.7	680	3.4
2	10	300°	0.7	660	3.2
3	10	310°	0.7	650	3.7
1	15	360°	0.7	700	3.8
2	15	340°	0.7	690	3.5
3	15	345°	0.7	680	4.1
4	15	350°	0.7	690	4.0

Another group of six APS 3-1200 phase shifters was also sprayed under slightly varied parameters. The differential phase shift, as a function of drive for these six phase shifters, is shown in Figure 18. These results further indicated that the reproducibility of the arc plasma process was good, and that a high yield of acceptable devices could be expected. A high yield is necessary in order to consider the arc plasma process acceptable from the

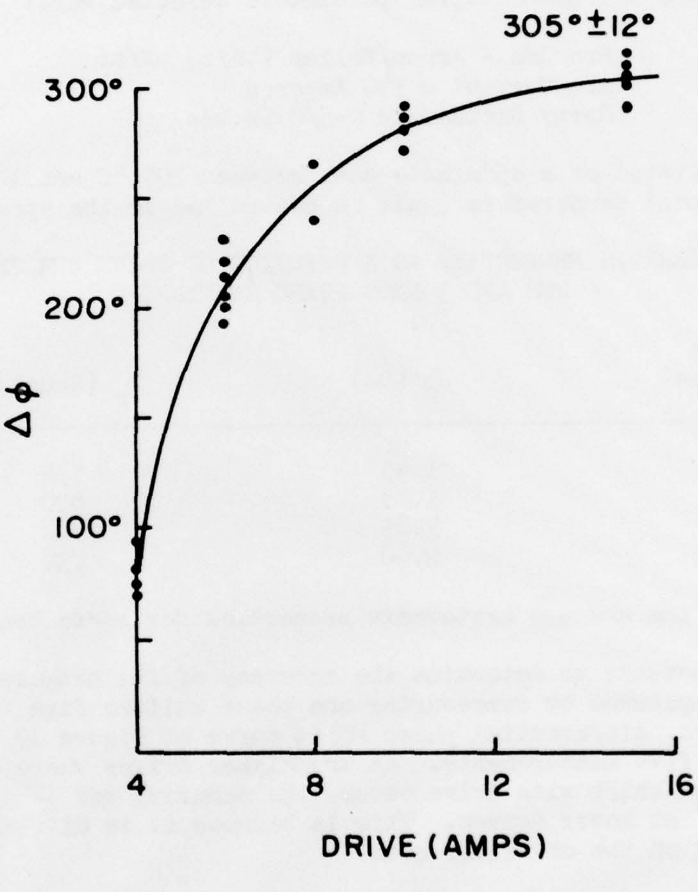

Figure 18. Original Reproducibility of APS Phase Shifters

standpoint of economy. The actual yield necessary has not yet been determined.

Encouraged by the above results, a set of ten phase shifters were fabricated over a three week time frame, using arc plasma spray parameters and anneal cycles as identical as possible. However, to simulate a much larger run, the arc gas tanks were changed and the powder, though similarly processed 3-1200, was taken from two different batches.

A non-magnetic lithium ferrite dielectric with an expansion coefficient of $14.6 \times 10^{-6}/^{o}C$ produced the best hysteresis results with this 3-1200 composition. The arc plasma spray parameters selected were:

Arc Gas - Argon/Helium (46/6) cf/hr
Arc Current - 340 Amperes
Spray Distance - 2-5/8 inches

The anneal consisted of a 45 minute soak between 1000°C and 1020°C; this was as close as anneal temperature could be controlled in the spraying oven.

TABLE 14. HYSTERESIS PROPERTIES AS A FUNCTION OF DRIVE CURRENT FOR APS 3-1200 PHASE SHIFTERS

Drive (Amperes)	H_c (Oe)	B_r (Gauss)
3	1.45	455
4	1.65	620
6	1.85	700
10	2.00	730

Table 14 shows the average hysteresis properties for these ten phase shifters.

It was necessary to determine the accuracy of the measurement technique, which was accomplished by remeasuring one phase shifter five times. The drive current vs. differential phase shift curve of Figure 19 shows the results of these five measurements. At the higher drives where only small changes of phase shift with drive occur, the accuracy was $\pm 4^{o}$, which decreased to $\pm 15^{o}$ at lower drives. This is because it is difficult to set the drive precisely on the dc driver used.

The determination of yield would be based on the phase shifter performance at 10 and 15 amperes. An acceptable differential phase shift variation would be less than $\pm 10^{o}$. This allows for $\pm 6^{o}$ greater than the measurement accuracy which is a tighter requirement than for most current phased array systems. Also the insertion loss would have to be less than 1.0 dB.

The ten arc plasma phase shifters were fabricated and machined. During machining, one phase shifter was misaligned and machined with 35 mil walls, as compared to 50 mil required. This phase shifter could not even be fixed in the test fixture, and though not due to the arc plasma process, this phase shifter was counted as a failure. The other nine phase shifters had

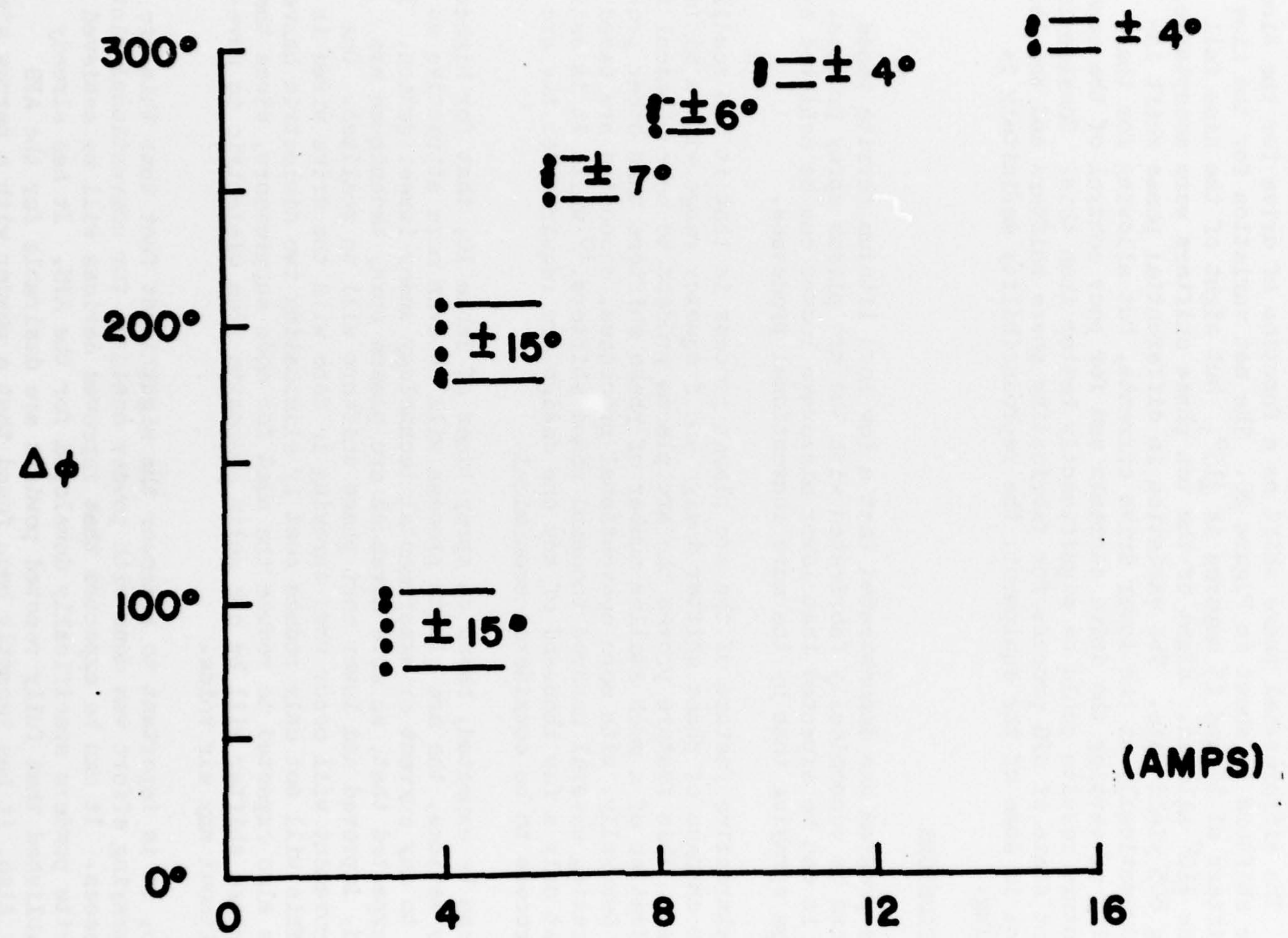

Figure 19. Accuracy of Differential Phase Shift Measurement

machining variations of $\pm$ 3 mil, i.e. $\pm$6%.

All but one of the nine APS phase shifters had insertion losses equal to or less than 0.5 dB. The other phase shifter had a loss of 0.7 dB, though from the accuracy measurements, insertion loss varied as much as 0.15 dB. The differential phase shift as a function of drive for the nine APS phase shifters is shown in Figure 20. The net variation for the nine phase shifters at 10 and 15 amperes is $\pm 15^{\circ}$; but eight of the nine fall within the $\pm 10^{\circ}$ allowed. Eight of the ten phase shifters were acceptable, producing 80% yield rate. The variation in differential phase shift increases dramatically at the lower drive currents, but allowing for the sensitivity of setting the drive currents and for poor control of the anneal cycle, future results could be significantly better than this. Considering the infant state of APS process for fabricating phase shifters and the lack of control in some of the equipment, the reproducibility definitely is encouraging.

CONCLUSIONS

This program has demonstrated that a low loss lithium ferrite phase shifter can be economically fabricated with the arc plasma spray process. Further, it can be expected that lower microwave losses can be achieved by arc plasma spraying than by the more conventional processes.

An attractive feature of the arc plasma process is that it can readily adjust to changes of phase shifter design and frequency range with a minimum of expense. This feature proves the arc plasma process to be economical for the fabrication of a much smaller number of phase shifters than other processes. Generally, with more conventional processes, economics are based on fabricating several hundred thousand phase shifters,[10] while it is estimated that only a few thousand of any one design are required for the arc plasma process to be considered economical.

It can be expected, based on spray times of Table 10, that for higher frequency devices, the arc plasma process will be even more attractive as compared to any current or experimental technology under investigation. It is also expected that, as more advanced arc plasma spray techniques are developed, improved and lower cost phase shifters will be realized. One such improvement will occur when spraying is done with the drive wires in place. This will not only reduce cost by eliminating two dielectric halves, but it is also expected to reduce the need for mode suppressors, since the ferrite phase shifter will be one solid composite from dielectric to waveguide, without any air voids.

Also, it is important to remember the significant fact that this arc plasma spraying effort was done with powder designed for conventional sintering processes. It can be expected that improved devices will be achieved with ferrite powders specifically developed for the APS. It has already been established that fully reacted powders are desirable for the APS process. Also, it has recently been found that a powder with a narrow size

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10. C. R. Boyd, Jr. "Comments on the Design and Manufacture of Dual-Mode Reciprocal Latching Ferrite Phase Shifters," IEEE Trans., Vol. MTT-22-6, pp 593, June 1974.

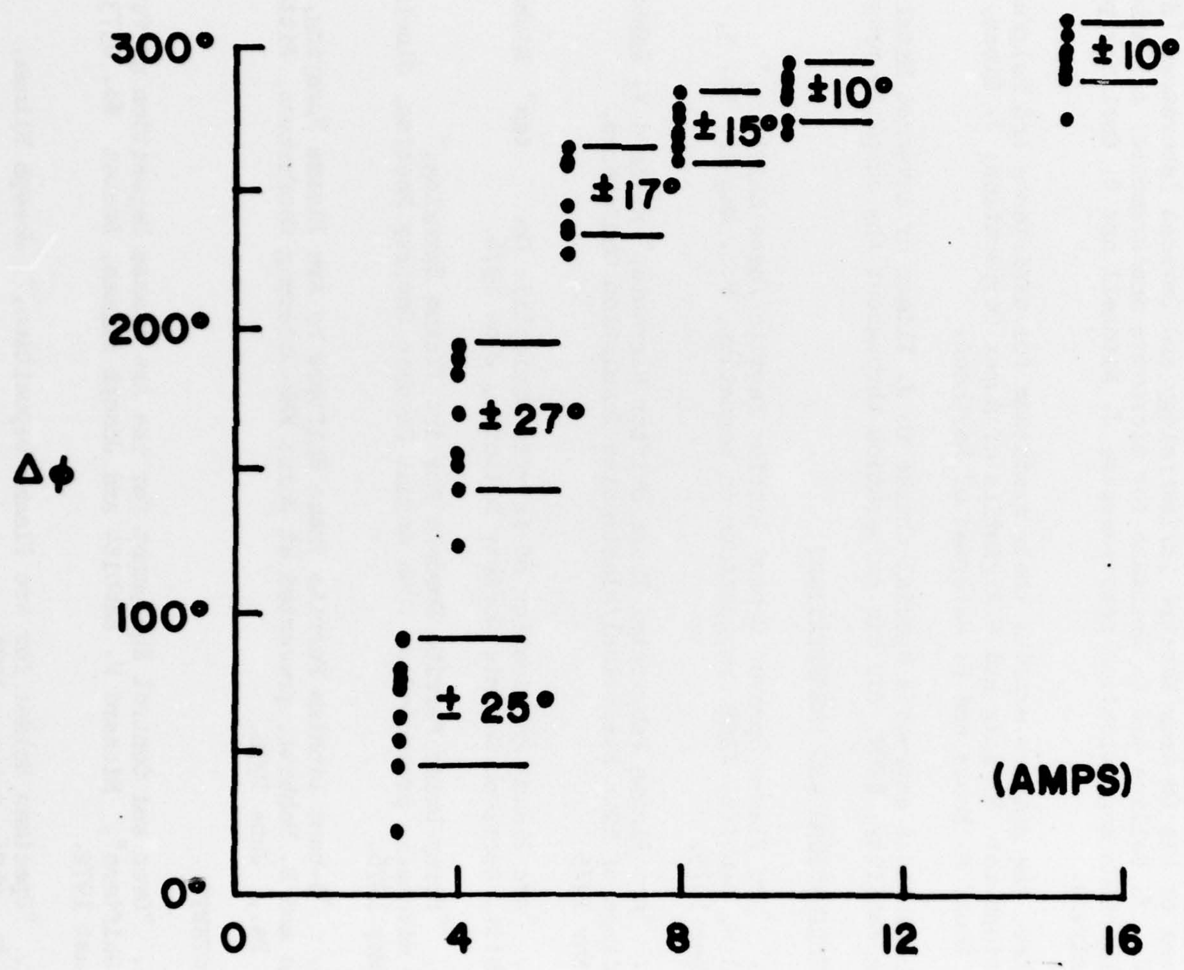

Figure 20. Reproducibility of the APS Process

distribution improves the reproducibility of the APS process. Neither of these two characteristics, i.e. fully reacted and narrow size range, are generally desirable for conventional sintering processes. Finally, the newer higher power and higher velocity arc plasma guns should also contribute to improved materials and devices from the arc plasma process.

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PUBLICATIONS AND PRESENTATIONS

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